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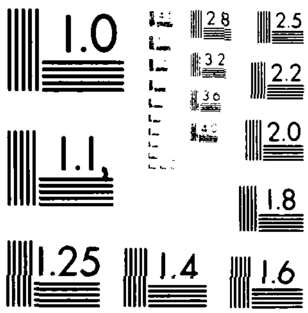

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ADVANCED CHEMICAL CHARACTERIZATION AND PHYSICAL PROPERTIES OF ELEVEN LUBRICANTS

INTERIM REPORT
AFLRL No. 166

By

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Additives	Chemical Characteristics	XRF Spectroscopy

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Lubricants used in the power trains of helicopter transmissions have required "standard tests", such as ASTM-designated methods, definitizing required physical and chemical properties. These initial properties and their subsequent service life variations or degradations, whether causation is internal or external, affect service performance. In order to advance the state-of-the-art lubricant "performance" predications, tests in the regimes of friction, wear, high-pressure viscosity, particulate

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20. ABSTRACT (Cont'd)

contamination and others are now part of "standard practice" techniques available. Along with these tests, a third generation chemical characterization technique has now been developed which is fast, efficient, accurate, and requires only milligram sample amounts with a minimum of processing. This technique yields accurate compositional data of ester-type lubricants and some antioxidant additives. Other metal-type additives are determined by spectroscopic methods while lubricant classifications are determined by a combination of Infrared Spectroscopy and boiling point distribution by gas chromatography.

FOREWORD

The work reported herein was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (AFLRL), located at Southwest Research Institute, San Antonio, Texas.

The initial phase covering the physical properties was conducted under NASA Purchase Requisition No. 520047.

The high-pressure viscosity, friction and wear tests, and chemical characterization work was conducted under Contract No. DAAK70-82-C-0001 during the period August 1982 through February 1983. The contracting officer's representative was Mr. F.W. Schaekel (DRDME-GL, MERADCOM).

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I. INTRODUCTION

Lubricants play a decisive role, having myriad critical operation "performance index" parameters in helicopter power train components. However, the performance of helicopter transmissions still requires advances in predictive analytical methods and tests so as to evaluate and compare lubrication effects on operational life, reliability, friction, wear, service overhaul schedules, build and operational cost effectiveness. The rapid scientific and technological advances in power transmission technology (1)* has markedly emphasized the need to evolve new lubrication evaluation techniques (2) at the fundamental molecular and chemical property levels. Also, basic research investigations are required on lubricant dynamical and chemical surface interactions including investigations under simulated field service environments

Typically, a lubricant is now chosen based on the specification by which it is qualified. Frequently, the specification contains the performance requirements for the lubricant. In field applications or in performance studies, the lubricant is thus selected based on the specification. In other cases, a lubricant is selected because it is classified as a lubricant for a given application. Seldom is the composition of the lubricant considered in its overall application selection. Comparison of the composition of different lubricants and correlation of lubricant performance as related to the chemical composition is difficult because insufficient specific lubricant composition information is available. As a result of the recent developments in lubricant analytical chemistry at the U.S. Army Fuels and Lubricants Research Laboratory (AFLRL)(3), lubricants can now be characterized as to their chemical composition.

Modern lubricants are complex chemical mixtures containing one or more basestock (major) components, and several additives that allow the finished lubri-

*Underscored numbers in parentheses refer to the list of references at the end of this report.

cant to perform its function in an engine or other power flow system. The lubricant basestock usually contains either:

(a) mineral oil (solvent neutrals, pale oils, bright stocks, etc.),
(b) synthetic hydrocarbon(s) (polyalphaolefins, polyalkylbenzenes, etc.),
(c) synthetic organic compounds other than hydrocarbons (mono-, di-, tri-, and tetra-esters, ethers, phosphate esters, polyol esters, polyethers, silicones, etc.), or
(d) a combination of the above.

Similarly, the additive package in a finished lubricant may have several constituents including detergent, dispersant, antioxidant, antiwear agent, extreme pressure additive and possibly a viscosity index improver. While some components may exhibit multifunctional properties (improve more than one function of the lubricant), the number of major constituents of a lubricant may be large indeed. Turbine engine lubricants and transmission fluids are specially formulated to meet these application needs.

Detailed compositional information is generally needed to define basestock character and to correlate the basestock component type to the families of refined lubricants, power train, and hydraulic fluids.

The compositional information needed generally takes the following form:

1) <u>Physical Data</u> - comprising those data needed for specifications, sometimes referred to as "Standard Tests", and necessary to develop correlations to performance.
2) <u>Friction and Wear</u> - standard test (ASTM D 2714) to aid in determining lubricating properties.
3) <u>High-Pressure Viscosity</u> - correlates to a lubricant's performance under actual operating conditions.
4) <u>Chemical Characterization</u> - supplies detailed information of the lubricant's actual chemical composition.

The physical data tests, friction and wear test, and high-pressure viscosity test methods are industry-accepted standard tests, and present no new areas

for defining or characterizing a lubricant. However, recent advances in the analytical chemistry of lubricants at AFLRL has allowed simpler and more accurate quantitative chemical characterization of the basestocks and some organic additives.

This study was undertaken to define the composition of basestock materials so that, ultimately, better correlations with the critical operational performance index parameters can be made. In the work described in this report, the composition of eleven lubricant basestocks has been determined.

II. OBJECTIVE

The objective of this program was to provide NASA-Lewis Research Center and the U.S. Army Aviation Research and Development Command (USAAVRADCOM) Research and Technology Laboratories with data concerning both the physical and chemical properties of eleven lubricants selected by NASA-Lewis engineers for performance evaluation as helicopter transmission lubricants (Table 1).

III. APPROACH

To accomplish that objective, a variety of fluid types were chosen, including MIL-L-23699, and MIL-L-7808 qualified lubricants, synthetic hydrocarbon-based oils, and two automotive-type automatic transmission fluids. Standard physical tests and wear metal analyses were conducted on both the new and used lubricants. In addition, boiling point distribution by gas chromatography, infrared spectrophotometric analysis, chemical characterization of each lubricant basestock by a newly applied derivatization/gas chromatographic techniques, high-pressure viscosity measurements as a function of temperature using a falling body viscosimeter, and friction-wear tests using an LFW-1 test machine were conducted. Tabulation of results and descriptions of the methodology applied are contained in the following sections.

TABLE 1. SYNTHETIC LUBRICANT ANALYSIS
SAMPLE IDENTIFICATION

NASA-Lewis Description	SwRI Oil Code	Specification	Type
A-New A-Used	AL-11252-L AL-11253-L	Dexron II GM 6137-M	Automatic Transmission Fluid
B-New B-Used	AL-11268-L AL-11269-L	Dexron II GM 6137-M	Automatic Transmission Fluid
C-New C-Used	AL-11250-L AL-11251-L	MIL-L-23699	Turbine Engine Oil
D-New D-Used	AL-11254-L AL-11255-L	MIL-L-23699	Type II Synthetic Gas Turbine Engine Oil
E-New E-Used	AL-11256-L AL-11257-L	Type I Synthetic Gear Lubricant	
F-New F-Used	AL-11258-L AL-11259-L	Syn. Hydrocarbon w/Antiwear Additives	
G-New G-Used	AL-11260-L AL-11261-L	MIL-L-2104C MIL-L-46152	Synthetic Fleet Engine Oil
H-New H-Used	AL-11262-L AL-11263-L	MIL-L-7808	Turbine Engine Oil
I-New I-Used	AL-11264-L AL-11265-L	MIL-L-23699	Type II Turbine Engine Oil
J-New J-Used	AL-11270-L AL-11271-L	MIL-L-23699	Type II Turbine Engine Oil
K-New K-Used	AL-11266-L AL-11267-L	Turbine Engine Oil	

IV. EXPERIMENTAL

A. Physical Data

The physical data for each oil were obtained by standard test methods shown in Table 2. The data are tabulated and presented in Appendix A.

TABLE 2. SYNTHETIC LUBRICANT ANALYSIS
METHODS USED IN ANALYSIS

Method	Reference
Kinematic Viscosity	ASTM D 445
Gravity Specific API	ASTM D 1481
Total Acid Number	ASTM D 664
Particulate Contamination Count	ARP 598 (Revised 8-1-69)
Wear Metals Tests	
X-ray Fluorescence-Filter Method	AFLRL Report No. 102*
Spectroscopic Analysis-WPAFB Acid Extraction Method (Mod)	AFWAL TR-80-4022*
Specific Heat, Differential Scanning Calorimetric Method	Section Six, DSC and Pressure* DSC Cells and Accessories/ Instruction Manual 990 Thermal Analyzer and Modules
Boiling Point Distribution Simulated Distillation	Modified ASTM 2887*

*These methods are described in detail in the text.

B. Friction and Wear Tests

In this program, it was mutually agreed that the eleven NASA-Lewis supplied lubricating oils were to be tested in duplicate on a LFW-1 friction and wear testing machine per ASTM D 2714 (1978) and modified as follows:

(a) Surface speeds to be 180 ft/min (54.9 m/min).

(b) Hertz line contact stress to be 100,000 psi.

(c) Block and ring material to be AISI 9310 (AMS 6260) steel with black oxide finish and with Rockwell C 60 hardness and 8 microinch surface finish.

(d) Test oil temperature to be 100°C (212°F) during testing.

(e) Test duration to be 10,000 cycles (ring revolutions) with friction force measured and recorded at 400, 800, 1200, 9000, and 10,000 cycles.

C. High-Pressure Viscosity

The viscosity as a function of pressure and temperature was measured in a falling body viscometer. The variable range for this instrument was 1 atmosphere (101.3 kPa) to 604 MPa in pressure, 20° to 150°C in temperature, and about 0.4 mPa·s to 1000 Pa·s in viscosity. The viscometer consists of a magnetic sinker in a nonmagnetic pressure vessel which is surrounded by a linear variable differential transformer. The viscosity measurement is made by timing the sinker fall over a predetermined and variable fall distance. The fall distance is varied depending upon the viscosity level. The sample is isolated from a pressurizing medium by a floating piston. The pressurizing medium, which is a low-viscosity diester, is pressurized by a hand-operated hydraulic pump operating through an intensifier. The intensifier has an area ratio of approximately 15 to 1.

The viscometer is housed in an air oven to control the temperature. The temperature is measured by a thermocouple inserted in a well in the pressure vessel. The pressure is measured by a Bourdon gage on the low-pressure side of the intensifier. The system pressure has been calibrated for seal friction in the intensifier and isolating piston. The viscometer fall constant as a function of pressure and temperature has been calibrated using 2-ethyl-hexyl-sebacate and the data obtained by P.W. Bridgman as reported in the ASME Pressure Viscosity Report. (4) A minimum sample size of 2 cubic centimeters is required. Further description of similar instruments, and data acquired with them can be found in References 5 through 7.

The data obtained by this method are shown in Appendix C.

The following definitions and conversions may be helpful:

$$\alpha_{OT} \equiv \left.\frac{d\ln\mu}{dp}\right|_{T,p=1\,atm} = \frac{1}{\mu}\left.\frac{d\mu}{dp}\right|_{T,p=1\,atm}$$

$$\alpha^* \equiv \left.\left\{\int_0^{p\to\infty} \frac{\mu(T,p = 1\,atm)}{\mu(p,T)}\,dp\right\}^{-1}\right|_T$$

α^* is a more reliable measure of the viscosity-pressure response of the material. It is determined by integration, employing all the data measurements, while α_{OT} is obtained by graphical differentiation and is very dependent on a few of the low-pressure data points; hence, it is subject to more overall error.

$$p/\text{psi} = p/\text{MPa} \times \left(\frac{10^6}{6.894 \times 10^3}\right) = p/\text{MPa} \times \left(\frac{10^3}{6.894}\right)$$

$$\mu/\text{cp} \equiv \mu/\text{mPas}$$

$$\mu/\frac{\text{lb}\cdot\text{s}}{\text{in.}^2} = \mu/\text{mPas} \times \left(\frac{10^{-3}}{6.894 \times 10^3}\right) = \mu/\text{mPas} \times \left(\frac{10^{-6}}{6.894}\right)$$

D. Analytical Characterization

Several analytical techniques and separation methods are referred to for the characterization of lubricants.(2) The utility, applications, and resulting data produced by the application of these techniques are discussed best when

segregated into specific analytical chemistry groups: (1) Spectroscopic methods, (2) Gas chromatography, and (3) Chemical derivatization.

1. Spectroscopic Methods

a. Infrared Spectrophotometry

When a lubricant is submitted for analysis, first an infrared (IR) spectrum is obtained. Application of IR spectroscopy is useful because it allows one:

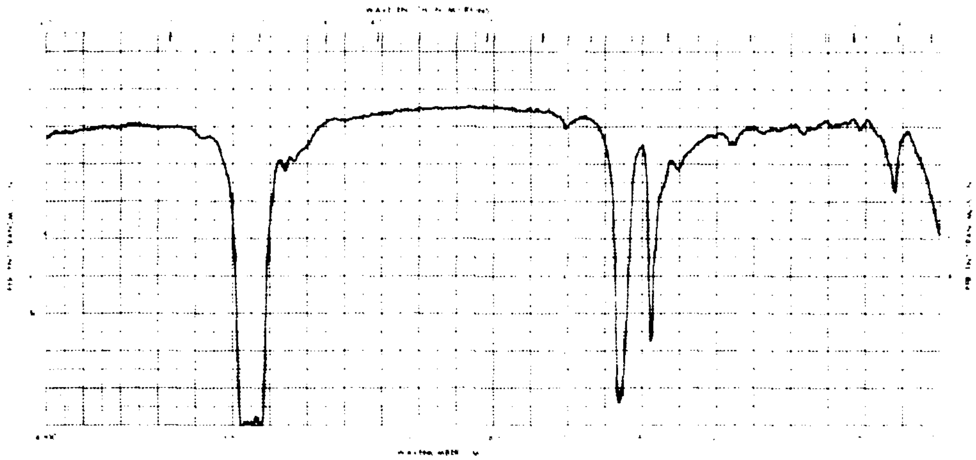
- to retain a permanent record of a given oil formulation that will serve as a basis for the detection of possible deviation from the originally approved formulation;
- to determine the nature of basestock (e.g., mineral oil, polyalphaolefin, ester, polyalkylated benzene or blends);
- to detect the presence of certain additives; and
- to detect the presence of oxidation products (if acrylate-type viscosity index improvers are not present and/or corrected for) in used oils.

Some characteristic (diagnostic) IR wavelengths used in oil analysis are:

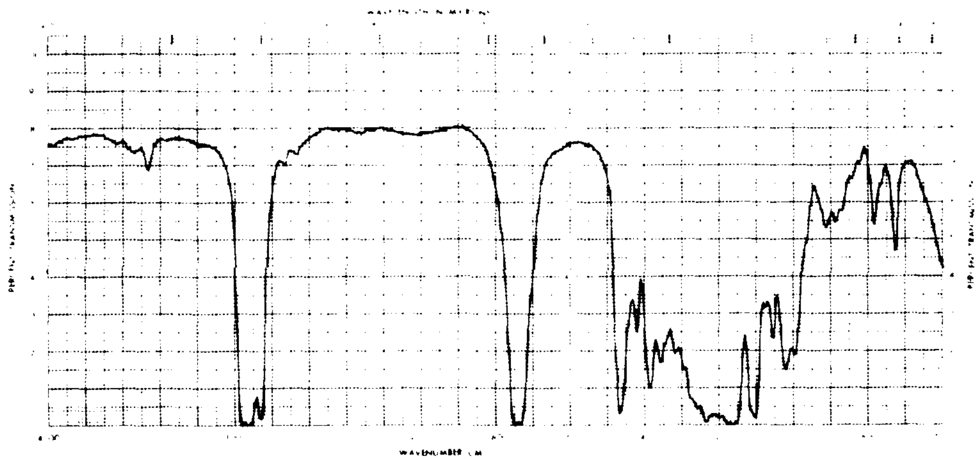
Frequency, cm^{-1}	Structure or Vibrational Mode Producing IR Absorption
3570-3200	OH stretching (e.g., glycols, phenols)
3500-3300	NH stretching (e.g., amines)
2960-2840	CH stretching (e.g., mineral oils)
1770-1650	C=O stretching (e.g., esters, some oxidation products)
1190-1160	C-O stretching (e.g., esters, ethers, alcohol)
1020-960	P-O-C (e.g., dialkyldithiophosphates)
1625-1575	Aromatic Ring Structure

A cursory IR spectrum of an oil, therefore, provides a wealth of information that is also used as a guide in the selection of the proper subsequent analytical methods.

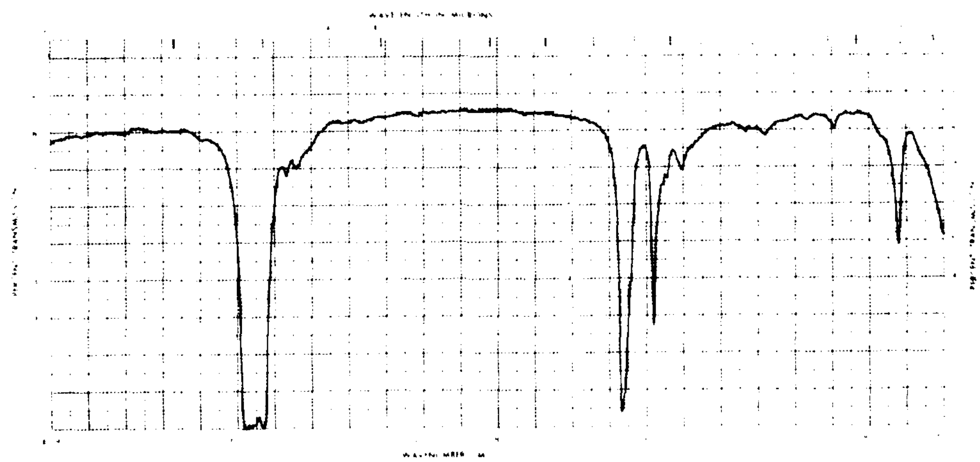
Figure 1 shows the spectra of a known petroleum hydrocarbon oil, a known ester type oil, and a known synthetic hydrocarbon (PAO) oil. Appendix G shows the spectra for the individual lubricant samples.



a. Petroleum Hydrocarbon Base Oil



b. Ester-Type Oil



c. Synthetic Hydrocarbon (PAO) Oil

FIGURE 1. STANDARD LUBRICANT-TYPE INFRARED SPECTRA

b. X-Ray Fluorescence Spectrophotometry

After an initial classifying IR spectrum is obtained on an unknown lubricant, usually a restricted elemental analysis is obtained. X-ray fluorescence spectrometry (XRF) is a convenient, fast, and nondestructive method capable of simultaneously detecting and quantitating elements from sodium (atomic number 11) up in the periodic system. Both metals and nonmetals, such as P and S, in lubricant additives, and wear metals in the case of used oils, are easily detected and measured without regard to the chemical form in which the elements are present. The minimum amount of element that XRF can measure depends upon the element in question, but is usually in the parts-per-million (ppm) range. A complete qualitative analysis o. a lubricant may take as little as two minutes. Since XRF analysis may take a sample in the form of a solid, liquid, or powder, sediments in used oils may be analyzed on a homogenized sample or as a simple filtrate.

Results of the X-ray analysis may be used to direct further investigation toward restricted areas, i.e., toward the analysis of specific additives or may be used as a completed answer when only wear or contaminant metals identification is desired.

As is the case for most spectroscopic analytical methods, XRF is also capable of "fingerprinting" products. If the "fingerprint" of two products are not identical, the products are not identical. Atomic absorption techniques also continue to be used to supplement X-ray to provide quantitative data for certain metals.

The XRF data obtained for the lubricants in question are tabulated in Table A-5, and notes on the XRF analysis of the subject lubricants are listed in Table A-6, contained in Appendix A. Figures A-1 to A-11 show the XRF spectra for each lubricant sample.

2. Gas Chromatographic Method for Boiling Point Distribution

In the overall purpose of this program, it was desired to characterize lubricants both qualitatively and quantitatively, for which gas chromatography

(GC) offers the greatest single instrumental-analytical capability. The general gas chromatographic approach taken was to use a method which eluted the sample as completely as possible (whether neat or pretreated lubricant) and to use as high an analytical elution temperature as feasible. For this reason, a method essentially equivalent to ASTM D 2887 (Test for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography) with a resolution of approximately 5.0 was used for the lubricants.

Boiling point distribution of mineral oils can be done both by molecular distillation and gas chromatography. GC not only has higher resolution, but is more accurate and less time-consuming than the molecular distillation approach. The GC approach assumes that the hydrogen flame ionization detector has essentially equal response for all hydrocarbons in the lubricant samples. Figure 2 shows the chromatograms for a known petroleum hydrocarbon oil, a known ester-type oil, and a known synthetic hydrocarbon (PAO).

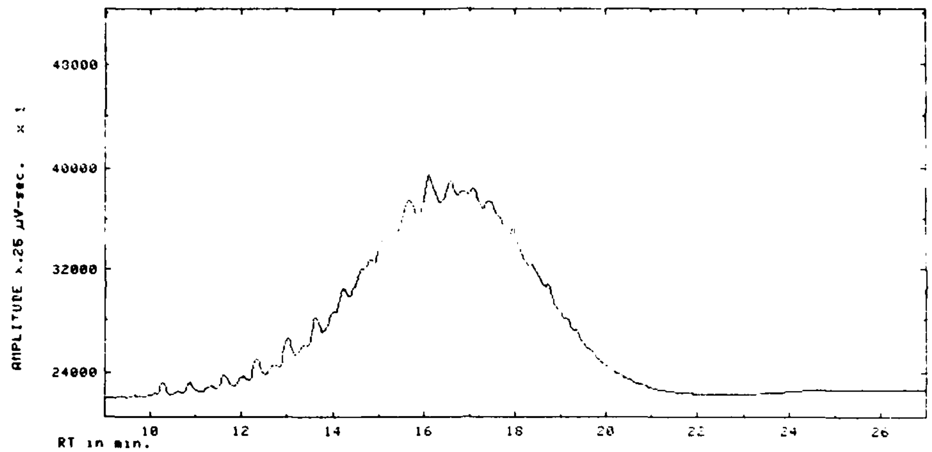
Initially, each oil was analyzed by infrared spectroscopy and compared to reference spectra (Figure 1) to determine its type. To confirm the type classification, the oils were analyzed by gas chromatography to determine their boiling point distribution (BPD) (Table D-1, Appendix D). Because the standard ASTM technique (8) for BPD has an upper temperature limit below that expected for the lubricants, an AFLRL modification allowing an extension of the upper temperature limit was used for this work.(9) The figures in Appendix D show typical chromatograms obtained for each lubricant type.

The petroleum hydrocarbon-based lubricants are adequately characterized by their boiling point distribution alone (AFLRL modification of ASTM D 2887), and no further characterization analysis was performed.

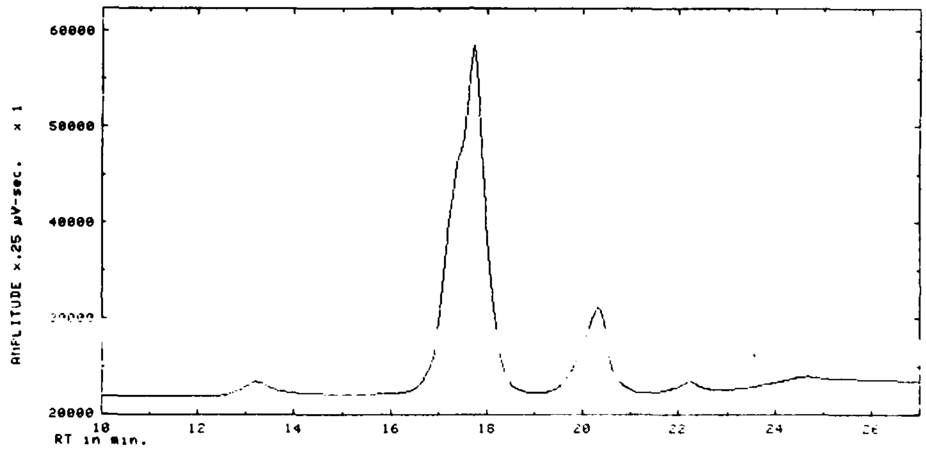
The composition of the PAO lubricants was characterized by comparing the peaks obtained from the BPD to hydrocarbon standards and known PAO lubricants analyzed under the same chromatographic conditions.

3. Chemical Characterization Methods

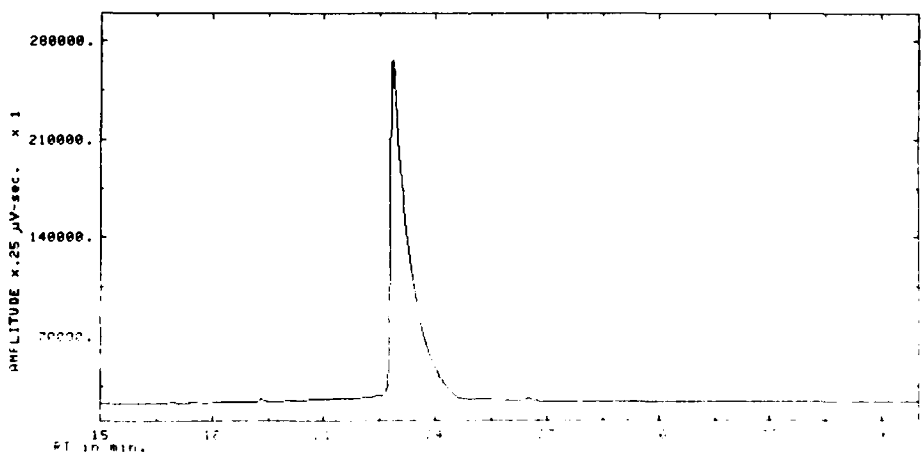
Further characterization of the ester-type lubricants necessitated identifying



a. Petroleum Hydrocarbon Base Oil



b. Synthetic Hydrocarbon (PAO) Oil



c. Ester-Type Oil

FIGURE 2. STANDARD LUBRICANT-TYPE CHROMATOGRAMS

the acids and the alcohols used to create the esters and quantitating these components. Techniques for characterizing the ester-type oils and those oils containing ester components were investigated to determine the most efficient method to use. (10-13) Transesterification techniques offered the most efficient method for analysis of the esters. Since no specific published transesterification techniques could be identified for lubricant-type esters, it was decided to approach this analysis with regard to the fact that the lubricants were esters and should be amenable to transesterification.

Much work has been done by others in the characterization of triglycerides, the triple esters of glycerol and long chain fatty acids, by transesterification techniques.(11-13) This transesterification involves the splitting of the ester bond which separates the fatty acid from the alcohol with the subsequent formation of the methyl ester of the fatty acids. The methyl esters of the acids are considerably more volatile than the acid themselves, allowing for ease of analysis by gas chromatography. The reactions take place in situ, usually at room temperature, with no additional chemistry necessary. The GC analysis is performed on the intact reaction mixture, with no extractions or additional treatment required. This technique works on the esters only, with no effect on any free acids that may be present, and is reported to yield quantitative conversions. If this technique could be applied to the analysis of ester-type lubricants, it would greatly improve the reliability of the attempts to characterize these lubricants, both new and used, and could aid in determining the oil breakdown mechanism. Certainly, this technique would be a significant improvement over the methods previously used which involved hydrolysis by reflux with alcoholic potassium hydroxide for several hours, then extractive separation of the alcohol from the carboxylic acid salt, followed by acidification and extraction of the carboxylic acid. The acids, thus recovered, were then derivatized for analysis. This older method required the use of a relatively large sample size to start with, and suffered from probable high sample losses during workup. Transesterification techniques, if successful in this application, could prove to be fast, efficient, and yield more accurate quantitative results than the above described method.

a. Ester Transesterification Technique

To accomplish the ester transesterification, the following techniques were employed:

To a capped 1-mL reactivial (Pierce Chemicals) vessel containing approximately 10 to 30 mg of ester-type lubricant was added 300 microliters (0.3 mL) of 0.2 normal methanolic (m-trifluoromethylphenyl)trimethylammonium hydroxide (METH-PREP II, Applied Science Laboratories). The reaction mixture was allowed to stand in a warm water bath, approximately 50°C, for 15 to 20 minutes with occasional shaking. Completeness of reaction was determined by observing a clear methanol layer. The polyols, pentaerythritol (PE), and dipentaerythritol (DPE) are insoluble in alcohol and form a precipitant lower layer. The trimethylolpropane (TMP) is soluble in methanol (Figure 3) so that in the case of a 100-percent TMP ester, no layering as precipitate is observed. Care must be exercised to keep any moisture or water from entering the reaction mixture since water will effectively kill the reaction. Completeness of reaction may be monitored by injecting 1 microliter of the top layer into the gas chromatograph at 15-minute intervals of reaction time until no further changes in peak sizes are measured.

After the reaction has been completed, usually 15-30 minutes, the sample is diluted to 1 mL with methanol and the top layer analyzed by gas chromatography. This analysis will show the fatty acids present and TMP, if any. In addition, two antioxidant additives may also be determined with this step, n-phenyl-alpha-naphthylamine (PANA) and p,p'-dioctyldiphenylamine (Figures E-4 and E-5 in Appendix E).

b. Polyol Silylation Technique

To determine the PE and DPE polyols, the top (methanol) layer is carefully removed and enough N,O-bis(trimethylsilyl) acetamide in silylation grade pyridine (TRI-SIL/BSA, Formula "P", Pierce Chemicals) is added to the reactivial to make 1 mL of sample. The sealed vial is placed in a water bath at 60°-70°C for approximately 15-30 minutes. When a single clear solution is observed, the reaction is complete, forming the silyl derivative of the polyol. An aliquot is injected into the gas chromatograph and analyzed for PE

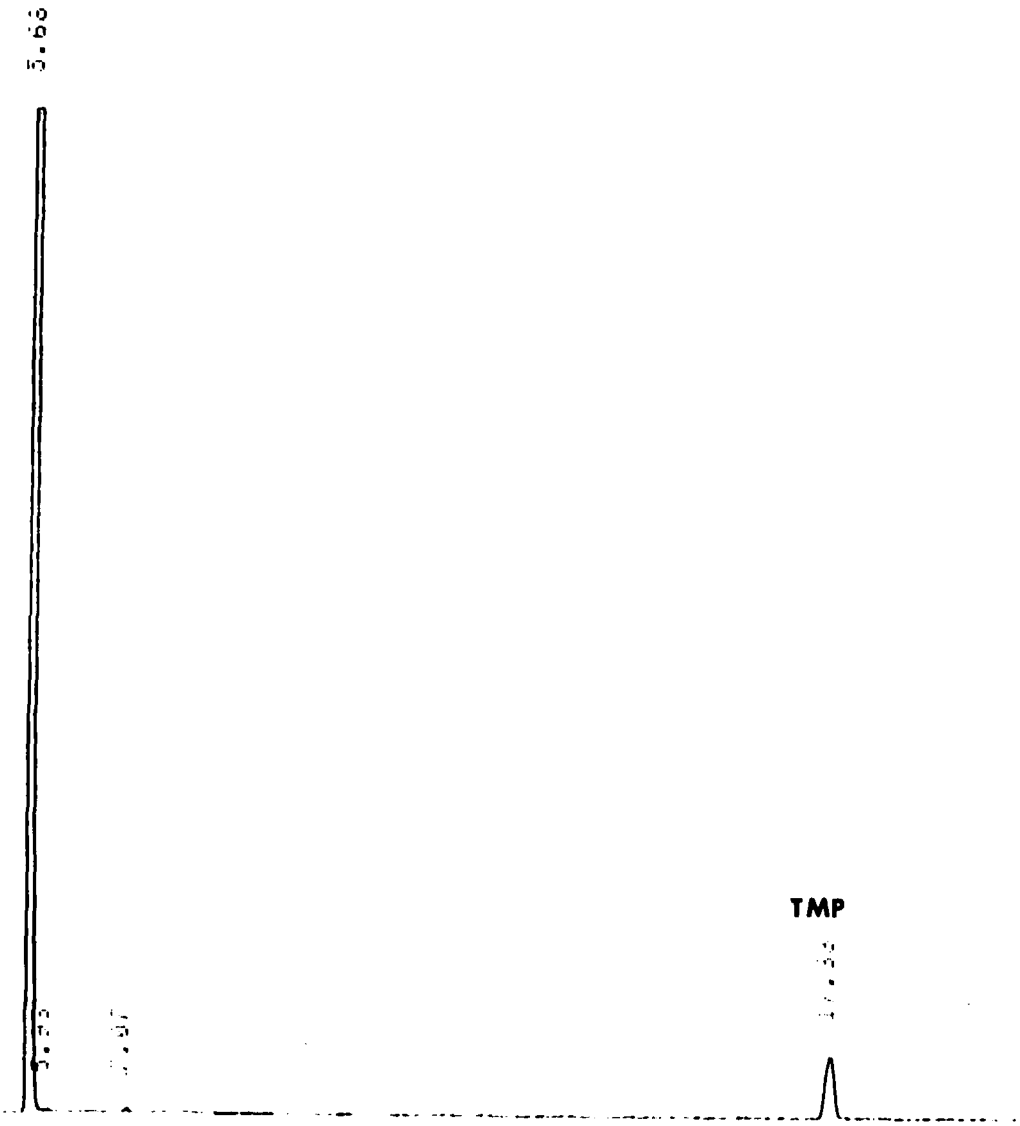


FIGURE 3. TMP IN METHANOL

and/or DPE. A small amount (0.05-0.1 mL) of the removed top layer may be added back to the lower layer prior to the addition of the silylating reagent to serve as markers for the chromatography.

V. DESCRIPTION OF METHODS

A. Wear Metals Tests

1. X-ray Fluorescence (Filter Method) (14)

In this method, samples are mixed well, and a portion is poured into a 50-mL beaker. The beaker is weighed, and most of the sample is poured into a 200-mL beaker containing about 50 to 60 mL filtered reagent grade heptane and stirred. The sample beaker is weighed again, and the sample weight is obtained by difference. The heptane-sample mixture is poured into a filtration apparatus designed to produce a 25-mm diameter deposit on a 0.45 μm Millipore filter. The filter is air dried and subjected to energy-dispersive X-ray fluorescence analysis.

With the particulate analyte on the filter separated from its fluid matrix, sensitivity is greatly enhanced. With this technique, 0.003 mg of each element may be detected. With sample weight of 30 grams, sensitivities of 0.1 ppm are possible. Wear metals, such as Fe, Ni, Al, and additive particulates which filter out of the oil, such as Ca and Ba, may be detected.

2. Spectroscopic Analysis for Iron

This method, evaluated in AFWAL-TR-80-4022, February 20, 1980, was modified to employ a visible light spectrophotometer to measure a colorimetric Fe reaction.

One mL of the oil sample is mixed with dilute H_2SO_4 and isoamyl alcohol. The acid serves to dissolve iron wear particles and aids in extracting the complexed iron from the oil. A buffer solution and a reducing solution are added to reduce all iron to the ferrous state. Addition of an indicator, bathophenanthroline disulfonic acid, forms a red-colored complex with the iron and is measured colorimetrically.

The modification made to the method described in the report involves the final dilution step. As described, 21 mL of iron-free distilled water is added to

the French square bottle used for the reaction, and the bottle is used as a cuvette in a Hach DR/2 spectrophotometer. Our reaction was conducted in 30-mL ground glass stoppered centrifuge tubes. The final dilution is performed with 15 mL of iron-free deionized water. An aliquot of the water phase is removed and measured in a 1-cm glass cuvette at 530 nm on a Beckman ACTA C III spectrophotometer. Standard solutions of 5-, 10-, 50-, and 100-ppm iron were used for calibration purposes. Iron concentrations of 1 ppm or more were easily detected.

B. Specific Heat by Differential Scanning Calorimetry

The oils were analyzed for heat capacity (Cp) values by differential scanning calorimetry (DSC). The instrument used was a du Pont Model 990. Both new and used oils were tested. The technique and calculations used were obtained from the du Pont Model 990 operating instructions. Instrument calibration was obtained using the heat of fusion of indium. Accuracy was determined using a sapphire standard.

1. Procedure

a. Background

Empty sample pans (and lids) were placed on the sample pedestal and repetitive traces made. Values for the periodic background data were plotted as a function of the time obtained. For final calculation, average or interpolated values for background were used. Absolute values for this measurement are arbitrary since a reference zero value is picked arbitrarily. This does not affect the final results, since it is the difference between measurements that is important.

b. Calibration

A sample of indium supplied with the DSC was weighed into an aluminum sample pan. The edges of the pan were then crimped and sealed. This sample was then run on a daily basis for most of the work. Using the known value for its heat of fusion and by determining the area of the endotherm using a polar plani-

meter, an average value for the cell calibration coefficient (E) was determined, using the following equation:

$$E = \frac{60\ AB}{H\ m} q_s$$

where

H = Heat of fusion (mcal/mg)

A = Peak area (sq in.)

q_s = Y-axis range [(mcal/sec)/in.]

m = Sample mass (mg)

B = Time base setting (min./in.)

c. Accuracy

(1) Temperature--Extrapolation of the leading edge of the above endotherm to the baseline yields the melting point of the indium sample. This may then be compared to the x-axis markers.

(2) Heat capacity--A specimen of sapphire was weighed and placed in a sample pan. Calculated values were compared with values determined for this material by the National Bureau of Standards.

d. Sample Introduction

Aliquots of the oil specimens were taken from the container as received. Aluminum sample pans were tarred prior to sample weight being recorded. The pan was then covered with an aluminum cover, and the assembly placed into the DSC.

e. Heat Capacity Measurement

The equation for calculation of heat capacity [using the calibration coefficient as determined in Sec. (2) above] was:

$$C_p(\text{mcal/mg deg C}) = \left(\frac{60E\Delta q_s}{H_r}\right) \frac{\Delta Y}{m}$$

where

- E = Cell calibration coefficient at the temperature of interest (dimensionless).
- Δq_s = Y-axis range, [(mcal/sec)/in.]
- H_r = Heating rate, (deg/min.)
- ΔY = Difference in Y-axis deflection between sample and blank curves at temperature of interest (inches).
- m = Sample mass, (mg).

f. Instrument Conditions

The following parameters were used to obtain the required data:

a. Starting temperature: 40°C (isothermal)
b. Ending temperature: 150°C (200°C for indium scan)
c. Program rate (after start): 10°C/min.
d. Recorder setting: 20°C/in.
e. Time rate (when used): 2 min./in.
f. Y-axis: 1 (mcal/sec)/in.
g. Average sample wt: 3 mg
h. Analysis temperatures: 84°C, 100°C, 140°C

2. Results

a. Instrument reproducibility

(1) Baseline--The standard deviation of 14 measurements was:

84°C	0.070 in.
100°C	0.079 in.
140°C	0.077 in.

The average deviation from the starting point was:

84°C	2.00 in.
100°C	2.00 in.
140°C	2.01 in.

(2) Indium--The standard deviations of 13 values taken at the above temperatures were 0.038, 0.034, and 0.037 in., respectively. Relative to the starting point, this is a variation of 1.8 percent, 1.6 percent, and 1.8 percent, respectively.

b. Calibration

The average peak area for 3.2 mg of indium over seven separate measurements was 0.195 sq in. with a relative standard deviation of 4.6 percent.

c. Accuracy

The table below shows the calculated and literature values for sapphire at the three temperatures of interest.

°C	Cp (calc.)	Cp (lit)
82	0.225	0.219 (380K)
100	0.227	0.225 (400K)
140	0.232	0.236 (440K)

d. Sample Data

The average values for heat capacity (Cp) and the standard deviation (σ) for each sample at each study temperature are presented in Table A-8 (Appendix A).

C. Gas Chromatography Methods

Two gas chromatographic methods are used for the analytical characterization of lubricants. They are discussed below.

Method 1 was developed at the AFLRL and has been in use in our laboratory for several years. It has been proposed for inclusion as an ASTM standard test method. Method 2 was developed in our laboratory specifically for this work.

1. Boiling Point Distribution of Lubricants (9)

The injection port for this system is an air-cooled 15.2 cm movable injector with a pyrex glass wool-packed metal port with a water-jacketed cooled septum. The sample is syringe injected into the glass wool, 6.4 cm from the septum face while the port is in the outer air-cooled position. The port is then pushed into a 7.6-cm heated jacket at 340°C, and after 3 minutes the port is pulled back to the air-cooled position. The water-jacketed inlet septum holder acts as a retainer when the port is pushed into the heated jacket. Approximately 8 cm of the injection port is in the heated jacket when the port is in the air-cooled position. This inlet hardware was designed to obtain the benefits of on-column plug injection but prevent contamination of the analytical column with the nonvolatile residual fraction of the sample. The water cooler prevents septum bleed at the elevated temperatures. Air cooling of the injection port reduces sustained vaporization (bleed) of heavy residual material in the sample. The movable injection port is connected to a 6 feet x 1/8-inch stainless steel coiled column in the column oven. A second column in the oven is used to provide dual column-dual detector (hydrogen flame ionization) operation to compensate for column bleed. The columns are packed with 10% Dexsil 300 on Chromosorb P, AW 45/60 mesh. The column oven is held at 0°C for 2 minutes and then programmed to 450°C at 15°C/min and held at 450°C for 5 minutes. An Altamont crude oil (obtained from the Bureau of Mines, Bartlesville, Oklahoma) diluted in carbon disulfide provides n-saturate peak identification to n-C_{60} (Figure D-1). Additionally, a special C_4-C_{40} normal saturate standard (Table 3) may be used for calibrating the Hewlett-Packard laboratory data system (Model 3354-B/C) boiling point distribution method.

2. Gas Chromatography of Lubricant Derivatives

A Hewlett-Packard Model 5880A capillary gas chromatograph equipped with a flame ionization detector (FID) and a 50 meter x 0.2 mm ID SE-54 fused silica capillary column was used for this work. The carrier gas was helium at a

TABLE 3. BOILING POINT DISTRIBUTION STANDARD
(Note: The following solution is diluted with carbon disulfide in the ratio 1:3.)

Carbon Number	Amount Per 100 ml
3	add to desired level
4	add to desired level
5	10.8ml
6	2.7ml
7	5.4ml
8	5.4ml
9	10.8ml
10	5.4ml
11	5.4ml
12	21.6ml
14	10.8ml
15	5.4ml
16	10.8ml
17	5.4ml
18	1.8g*
20	1.8g
24	1.1g
28	0.7g
32	0.7g
36	0.7g
40	0.4g

Injection volume is 2 microliters.
*C_{18} to C_{40} are solids.

nominal flow rate of 1.0 mL/min. The FID was maintained at 400°C and the injector at 375°C. A split injection technique was used at a split ratio of 100:1 with a 1.0 microliter injection. The oven temperature was programmed from 30° to 320°C at 10°C per minute with a final hold of 16 minutes. A calibration standard of the mono-carboxylic acid methyl esters from n-C_4 to n-C_{10} (Figure E-1), and mixed dicarboxylic acid methyl ester standard from n-C_5 to n-C_{10} (Figure E-2) were prepared. In addition, the silyl derivatives of TMP, PE, and DPE were prepared for calibration use (Figure E-3). Derivatives of the two antioxidants PANA (Figure E-4) and p,p'-dioctyldiphenylamine (Figure E-5) were prepared by the transesterification technique applied to the ester-type lubricants. Response factors for all compounds analyzed using the FID was set at 1.00. Compounds were identified by comparison of their retention times to that of the standards. Figure E-6 shows the results of the transesterification and silylation technique applied to sample AL-11250-L (NASA C).

VI. DISCUSSION

This report presents physical and analytical chemical characterization data for the eleven lubricants which were used in transmission performance studies by NASA-Lewis engineers. No field hardware performance tests were conducted by this laboratory and, at the request of NASA-Lewis, there was no attempt to correlate these data with the lubricants' field performance. The correlation of the chemical and physical data to the performance of the lubricants is outside the scope of this report but will be discussed by NASA-Lewis engineers in a separate NASA-Lewis report.

A. Physical Test Methods

1. Specific Heat

From the data shown in Table A-8, it can be noted that the differential scanning calorimetry (DSC) instrument, when run with either a blank or the standard material over again, has good precision and accuracy. However, when the sample oils were introduced, precision became quite poor, particularly for the "used" oils. Therefore, it is felt that the samples are probably not homogeneous. Thus, without prior filtering or some appropriate homogenizing treatment being performed, multiple values must be obtained and averaged to produce an acceptable value.

2. Friction and Wear Tests

Using the test conditions detailed in Section IV.B., the initial determination was attempted employing lubricant AL-11250-L (NASA Code C). In less than 100 cycles of the rotating test ring, contact seizure resulted, and the test block sustained gross asymetrical wear. The test was immediately repeated using the same test conditions and lubricant. Contact seizure between the block and ring again resulted at approximately 2250 cycles. It was then decided to employ lubricant AL-11266-L (NASA Code K), which is known to have a high load-carrying capability, and to attempt another LFW-1 test using the same test conditions as above. This lubricant successfully completed the test of 10,000 cycles, although there was a transfer of material from the block to the

rotating ring specimen as evidenced by the weight change at test termination. Based on these results, it was decided to try another lubricant under these same test conditions. Therefore, a test using AL-11252-L (NASA Code A) was initiated, and contact seizure resulted at approximately 1625 cycles. In view of these happenings and also due to the fact that AL-11250-L is a qualified MIL-L-23699 lubricant with demonstrated satisfactory performance in gas turbine engines, it was concluded that a 100,000-psi stress would be too severe in LFW-1 testing. Continued evaluation at that load would essentially provide little more than pass/fail results. This information was conveyed to the NASA-Lewis project engineer who approved of the recommendation to utilize a reduced machine load. Therefore, the test series was performed at a selected load to give an initial mean Hertz compressive stress of 80,000 psi. It is interesting that one more contact seizure was experienced employing lubricant AL-11250-L even at the reduced contact stress. It is also of interest that all tests having contact seizures both at the original load and at the reduced load resulted in a weight gain for the test ring, indicating a transfer of material from the stationary block to the rotating ring during testing. Normally, as expected, there was a weight loss for both block and ring during tests not experiencing seizure. After the problems discussed above were dealt with, the test series proceeded without difficulties. Appendix B presents test summary data for the 11 lubricants tested in accordance with the modified procedure.

B. Spectroscopic Methods

1. Infrared Spectrophotometry (IR)

IR offers a quick, easily interpreted method for identification of lubricant basestocks. The spectrum (Figure 1) for ester-type basestocks shows a prominent specific peak at 1730-1750 cm^{-1} which is absent from the spectrum of hydrocarbon oils. When compared to the synthetic hydrocarbon and petroleum hydrocarbon basestocks, the basestock type is quite evident. While the synthetic hydrocarbon and petroleum hydrocarbon spectra appear the same, which is expected since they are both essentially pure hydrocarbons, there is a signi-

ficant difference. The small peak at 1600 cm^{-1} in the spectra for petroleum hydrocarbons is due to aromatic hydrocarbon ring structure. This is typical for petroleum hydrocarbon basestocks and is not found in the synthetic hydrocarbon basestocks. The spectrum for the synthetic hydrocarbon basestock has no absorption peak at this frequency. Appendix G shows the spectra for the individual lubricant samples.

2. Metals Analysis

The X-ray fluorescence method for metals analysis offers a rapid, nondestructive, sensitive, and accurate identification and measurement technique for most metals found in lubricants. The use of the spectroscopic analysis for iron afforded an even greater degree of sensitivity when it was required. These data are presented in Tables A-5 through A-6. The interpretation of the XRF data is detailed in Table A-6.

C. Boiling Point Distribution by Gas Chromatography

The BPD method used for lubricants at the AFLRL is a modification to the ASTM D 2887 method. The modification enables the extension of the upper temperature limit as defined by the ASTM D 2887 procedure. This modification is presently being evaluated by the ASTM as a new method for inclusion in their list of standard methods. The chromatograms in Appendix D which this method produces show very distinctly different "patterns" for each type of lubricant basestock. Indeed, the patterns, especially for the ester-type lubricants, are virtual "fingerprints" for each sample and confirm the IR results.

D. Chemical Characterization

As an integral part of the Army's overall power train lubrication research effort, the AFLRL has been involved in developing the technology to characterize lubricants. The first generation approach to the analysis of lubricants was detailed in an AFLRL interim report published in March 1976.(2) Further developments and refinements led to a second generation analytical approach to the characterization of lubricants.(3,10,15)

This report details the third generation analytical approach to the analysis of lubricants. This approach has simplified the analysis of the lubricants by a rapid and easily accomplished in-situ derivatization of the esters by a transesterification technique. The GC analysis is conducted on the reaction mixture and yields detailed information regarding the chemical composition of the lubricants. In addition, the chromatography makes it possible to determine the presence of some organic antioxidant additives. Also, it may be possible to determine the causes of corrosion within the engine, and the reason for the corrosion variability between oils, if any. The technique utilizes very small sample amounts with a minimum of chemical treatment and handling. The results achieved using this new, third generation approach to the characterization of lubricants are summarized in Table 4.

The initial infrared spectroscopic examination of the lubricants provided a preliminary chemical class identification of each lubricant, i.e. petroleum, synthetic hydrocarbon, ester (Figure 1 and Appendix G). Coupled with the boiling point distribution chromatograms (Appendix D), the class or type identification proved to be positive in every case. Each type of lubricant yielded a distinctive chromatographic pattern (Appendix D). The type classification was further confirmed by the detailed derivative characterization work (Appendix F). Table 4 summarizes the basestock characterizations. The data in Table 4 are repeated in Table F-1 for convenience when referring to Appendix F.

TABLE 4. BASESTOCK CHARACTERIZATION SUMMARY

NASA Code	A	B	C	D	E	F	G	H	I	J	K
AFLRL Code	11252	11268	11250	11254	11256	11258	11260	11262	11264	11270	11266
Carboxylic Acids - %	Petroleum-based	Petroleum-based									
C-4			T	9						T	T
C-5			46	18	di-63				13	22	22
C-6			10	13	di-37			T	2	14	16
C-7			17	16			73	50	19	21	24
								35			
C-8			10	24			27	1	30	8	8
C-9			13	16				7	4	23	29
C-10			4	4				5	32	12	1
C-12								2	T		
Alcohols											
TMP							100	100	50		
PE			100	100					50	100	99
DPE											1
MONO-					(C13) 100						
Basestock Type											
Ester			x	x	dibasic		(20%)	x	x	x	x
Petroleum	x	x									
Synthetic Hydrocarbon						x	(80%)				
C30, %						43	38				
C40, %						45	50				
C50, %						12	12				

The chemical composition data for the test lubricant basestocks have also been presented graphically as a "Daisy Graph". A "Daisy Graph" is a method for representing a large number of parameters or variables in a simple fashion for easy comparison. The turbine engine oil "Daisy Graph" key is provided in Figure F-1. The angular position of the radial line is characteristic for each individual component. While not necessary, different colors have been used to illustrate the different chemical families of compounds for ease in comparing the composition of the lubricants. Red represents the mono-carboxylic acids present in polyol esters and blue represents the polyol base for the polyol ester. Green represents the mono-alcohols of dibasic acid esters, and black represents the base dicarboxylic acid of the dibasic acid ester. The length of the Daisy lines is proportional to the concentration of each component. In summary, the daisy key is outlined as follows:

Parameter/Color	Indicates
Red	Mono Carboxylic Acids
Blue	Polyols
Green	Mono Alcohols
Black	Dicarboxylic Acids
Length	Concentration

Following the analytical characterization scheme shown in Figure 4, two lubricant samples were identified as petroleum basestock types, AL-11252-L (NASA A) and AL-11268-L (NASA B) (Tables F-2 and F-3). They were characterized by comparison of their infrared spectra and boiling point distribution (BPD) chromatograms to those of known basestock types. Figure 1a shows the IR spectrum of a known petroleum hydrocarbon basestock lubricant. The major bands at 2800-3000 cm^{-1}, 1520 cm^{-1}, and 1370 cm^{-1} wavenumbers are due to C-H and CH_3 and are what would be expected for this type of material. When this spectrum is compared to the spectrum for a synthetic hydrocarbon (PAO) (Figure 1c), they appear almost identical with one important distinction. The small band at 1600 cm^{-1} wavenumbers is only seen for the petroleum basestock and is due to aromatics. The synthetic hydrocarbons (PAO) are composed of oligomers made by polymerizing an olefin, e.g., decene (C_{10}), to form compounds consisting of multiples of this C_{10} olefin, e.g., C_{20}'s C_{30}'s, C_{40}'s, and contain no aromatics. Therefore, the band at 1600 cm^{-1} is not seen in the spectrum for a PAO lubricant.

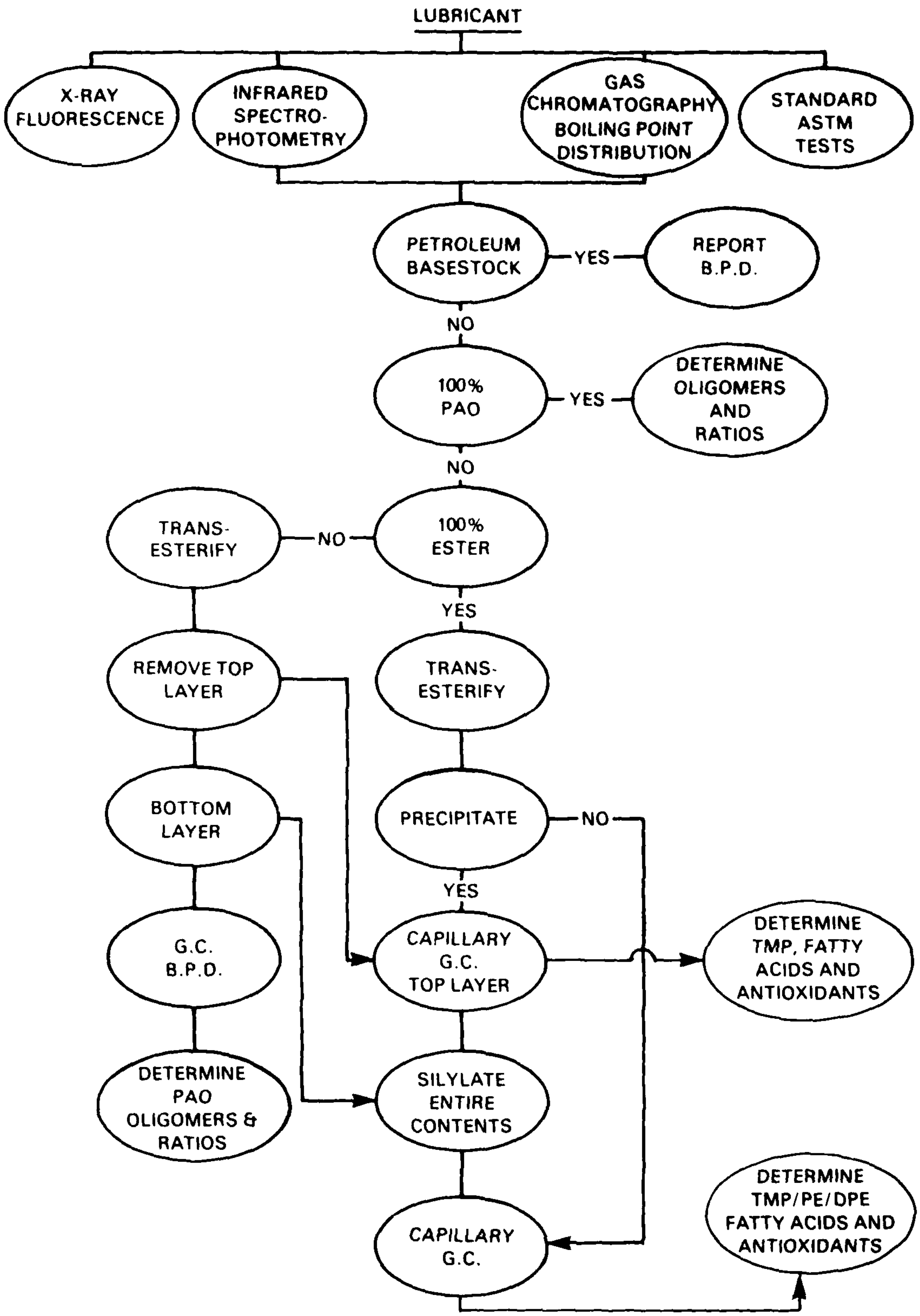


FIGURE 4. ANALYTICAL CHARACTERIZATION SCHEME

When the spectra for NASA samples A and B (Figures G-1 and G-2) are examined and compared to the known lubricant spectra (Figure 1), it becomes apparent that these samples are petroleum basestocks because of the typical C-H and CH_3 bands and the small band at 1600 cm^{-1} typical for aromatics. The small bands at 1700 cm^{-1} and 1735 cm^{-1} seen on both sample spectra are probably due to additives such as viscosity improvers, antioxidants, etc.

When the hydrocarbon spectra are compared to the spectrum of the ester-type lubricant (Figure 1b), the dissimilarities become apparent. The major band at 1740 cm^{-1} in the ester lubricant is due to the ester or C=O bands and is quite strong and specific. It is not seen in the petroleum spectra.

Figure 2 shows the chromatograms obtained for known petroleum, PAO, and ester-type oils. Each type yields a distinctively different chromatogram. The broad almost Gaussian-shaped hump of the petroleum oil (Figure 2a) is due to the large number of similar compounds emerging very close together and, when compared to the boiling point standard, its BPD may be easily determined.

The PAO chromatogram (Figure 2b) is characterized by the distinct separation of the oligomer groups. The carbon number range of each oligomer group may be determined by comparison to a boiling point standard (Figure D-2, Appendix D), which consists of known compounds eluting in boiling point order.

The chromatogram of the ester-type oil is a "fingerprint" pattern (Figure 2c). It is quite distinctive when the polyol esters cover a relatively broad range of esterified acids, e.g., C_4 to C_{10} monocarboxylic acids. If the esters should be of the dicarboxylic acid type, AFLRL experience has shown that they usually are very narrow in molecular weight range, e.g., C_7 and C_8 dicarboxylic acids and yield a chromatogram showing a relatively narrow well-resolved single peak (Figure D-6). By comparing the chromatogram and IR spectrum of an unknown sample to the chromatograms and IR spectra of the above described known oils, the basestock type and whether or not it is a blend of oil types can be determined.

NASA samples A and B were identified as petroleum basestocks by this technique, and no further chemical characterization was done (Appendix D, Figures D-4 and D-12).

From its IR spectrum and boiling point distribution chromatogram, one lubricant sample was identified as a 100 percent synthetic hydrocarbon type (PAO), AL-11258-L (NASA F) (Figure D-7). The very typical PAO chromatogram indicated that this lubricant was not a blend. The molecular weight range of the oligomers was identified by comparison to the calibration standard (Figure D-2) used for the boiling point distribution and to "standard" PAO lubricants of known composition (Table F-7).

One lubricant sample AL-11260-L (NASA G) (Figure D-8) was identified both by its infrared spectrum and BPD chromatogram as being a mixture of PAO and ester-type basestocks. Following the analytical characterization scheme, the entire sample aliquot was transesterified. This yielded two distinct layers. The larger, upper methanol layer containing the fatty acid methyl esters (FAME) was carefully separated from the lower layer and analyzed by capillary GC to determine the FAME composition. By comparison of the FAME analysis to the methyl ester standards (Figures E-1 and E-2), it was determined that the ester portion of this lubricant was composed of C_7 and C_8 monocarboxylic acids and TMP. The "Daisy Graph" (Figure F-5) shows the distribution graphically, and Table F-8 lists the values. The lower layer was chromatographed according to the BPD procedure. This yielded a chromatogram typical for a PAO. The PAO oligomers and their ratios were determined by comparison to the BP standard and the known PAO materials. In addition, the lower layer was silylated and analyzed by capillary GC for the presence of any PE and/or DPE.

Analysis of lubricant AL-11262-L (NASA H) (Figure D-9) showed a 100 percent TMP ester-type basestock with the carboxylic acids ranging from C_6 to C_{12}. The "Daisy Graph" (Figure F-6) shows the ratio of the components, and Table F-9 lists the actual values.

The IR spectra and BPD chromatograms of lubricants AL-11250-L (NASA C) (Figure D-3), AL-11254-L (NASA D) (Figure D-4), and AL-11270-L (NASA J) (Figure D-13) indicated a 100 percent ester-type basestock. The transesterification of these lubricants produced a precipitate. The capillary GC analysis of the supernatant layer showed a composition of FAME ranging from C_4 to C_{10}. Following this analysis, the entire transesterified sample was reacted with the silylating reagents to derivatize the precipitate. Analysis by capillary GC

of this mixture showed that these lubricants were 100 percent PE ester-type basestocks. The "Daisy Graphs" (Figures F-2, F-3, and F-8) show the ratio of the components, and Tables F-4, F-5, and F-11 list the actual values for each lubricant, which differ for each lubricant.

Analysis of lubricant AL-11264-L (NASA I) (Figure D-10) showed it to be composed of TMP and PE ester type basestocks, at a 50/50 mixture with the carboxylic acids ranging from C_5 to C_{12}. The "Daisy Graph" (Figures F-7) shows the ratio of the components, and Table F-10 lists the actual values.

Analysis of lubricant AL-11266-L (NASA K) (Figure D-11) showed a mixture of predominantly (99 percent) PE ester basestock with a small amount (1 percent) of DPE ester basestock and carboxylic acids ranging from C_4 to C_{10}. The "Daisy Graphs" (Figure F-9) show the ratio of the components, and Table F-12 lists the actual values.

VII. CONCLUSIONS AND RECOMMENDATIONS

The results of this new third generation analytical approach to the characterization of lubricants clearly indicate that it can be utilized as an established technique. It provides a quick and efficient route to the qualitative and quantitative determination of lubricant composition, not only for the basestock, but also for some organic additives, previously not easily amenable to analysis in fully formulated lubricants.

The application of newly developed chemical techniques plus the use of capillary column gas chromatography has greatly enhanced AFLRL capability to provide reliable and accurate information on lubricant composition. It is recommended that additional work continue towards reducing this technique to practical application.

With regard to the specific heat determination, it is recommended that additional testing continue on filtered lubricants, both new and used, to optimize the results of this technique. Because of the presence of particulate matter in a nonhomogeneous mixture, precision was poor, and multiple values had to be obtained to produce an acceptable average value.

During the LFW-1 friction and wear testing, some areas for improvement in testing surfaced.

Based on this work, the following additional activities are recommended to better characterize the above discussed lubricants:

- Perform additional LFW-1 friction and wear testing to determine if optimum test conditions such as temperature, load, wear specimen material, etc., can be established to better characterize the friction and wear properties of the lubricants and their basestocks.

- Perform ball-on-cylinder machine (BOCM) tests for comparison with the LFW-1 test machine results. The BOCM in its present configuration is primarily employed for fuel lubricity evaluations and would need to be modified with a higher temperature capability for lubricating oil evaluations. This machine is being widely used by CRC, commercial organizations, and testing laboratories for lubricity work both in this country and abroad.

- Since considerable engineering interest is being expressed in traction drives for new helicopter power systems, the methodology developed in this program should be applied to traction fluids. These fluids are chemically different from the petroleum oils, synthetic PAO's, and synthetic esters analyzed in this program. Traction fluids have special physical properties resulting from the unique chemical structures of the composite compounds employed. A major type of structure reported to be used in traction fluids is hydrogenated copolymers of α-methyl styrene and butadiene. These compounds exhibit reversible semi-solidification under extreme pressure and shear. The current methodology should be applied to compounds of this and other types to determine where it is useful and should be expanded to provide the necessary compositioned information in those areas where the need exists.

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APPENDIX A

PHYSICAL TEST DATA

TABLE A-1. ANALYTICAL REPORT
SYNTHETIC LUBRICANT ANALYSIS

NASA-Lewis Description	SwRI Oil Code	Viscosity @ Listed Temperature, cSt 40°C	82°C	100°C
A-New	AL-11252-L	37.48	10.48	7.01
A-Used	AL-11253-L	34.84	9.73	6.51
B-New	AL-11268-L	33.15	9.64	6.52
B-Used	AL-11269-L	31.79	9.21	6.24
C-New	AL-11250-L	26.40	7.69	5.13
C-Used	AL-11251-L	26.28	7.69	7.01
D-New	AL-11254-L	26.17	7.50	5.00
D-Used	AL-11255-L	26.12	7.49	4.99
E-New	AL-11256-L	33.91	8.91	5.87
E-Used	AL-11257-L	33.70	8.89	5.85
F-New	AL-11258-L	28.01	8.15	5.36
F-Used	AL-11259-L	27.98	8.04	5.35
G-New	AL-11260-L	56.65	15.05	9.83
G-Used	AL-11261-L	51.13	13.75	8.96
H-New	AL-11262-L	13.16	4.73	3.38
H-Used	AL-11263-L	13.05	4.65	3.32
I-New	AL-11264-L	24.19	7.18	4.85
I-Used	AL-11265-L	23.88	7.11	4.82
J-New	AL-11270-L	24.76	7.23	4.89
J-Used	AL-11271-L	24.60	7.20	4.88
K-New	AL-11266-L	26.39	7.61	5.09
K-Used	AL-11267-L	25.17	7.50	5.04

TABLE A-2. ANALYTICAL REPORT
SYNTHETIC LUBRICANT ANALYSIS

NASA-Lewis Description	SwRI Oil Code	Specific Gravity @ Listed Temperature, 40°C	82°C	100°C	API Gravity
A-New	AL-11252-L	0.8620	0.8558	0.8514	29.8
A-Used	AL-11253-L	0.8622	0.8544	0.8533	29.8
B-New	AL-11268-L	0.8626	0.8548	0.8546	29.9
B-Used	AL-11269-L	0.8625	0.8545	0.8552	29.9
C-New	AL-11250-L	0.9973	0.9862	0.9843	8.2
C-Used	AL-11251-L	0.9966	0.9880	0.9844	8.2
D-New	AL-11254-L	0.9868	0.9768	0.9746	9.7
D-Used	AL-11255-L	0.9867	0.9773	0.9745	9.7
E-New	AL-11256-L	0.9322	0.9211	0.9201	17.7
E-Used	AL-11257-L	0.9305	0.9215	0.9205	17.9
F-New	AL-11258-L	0.8262	0.8108	0.8088	36.0
F-Used	AL-11259-L	0.8244	0.8150	0.8139	36.3
G-New	AL-11260-L	0.8629	0.8536	0.8527	29.6
G-Used	AL-11261-L	0.8626	0.8517	0.8532	29.7
H-New	AL-11262-L	0.9442	0.9320	0.9313	15.7
H-Used	AL-11263-L	0.9438	0.9348	0.9307	15.8
I-New	AL-11264-L	0.9659	0.9568	0.9546	12.8
I-Used	AL-11265-1	0.9659	0.9566	0.9544	12.8
J-New	AL-11270-L	0.9856	0.9759	0.9747	10.1
J-Used	AL-11271-L	0.9856	0.9765	0.9747	10.1
K-New	AL-11266-L	0.9829	0.9721	0.9725	10.3
K-Used	AL-11267-L	0.9824	0.9755	0.9718	10.3

TABLE A-3. ANALYTICAL REPORT

SYNTHETIC LUBRICANT ANALYSIS

NASA-Lewis Description	SwRI Oil Code	Total Acid Number (mg KOH/g)
A-New	AL-11252-L	0.54
A-Used	AL-11253-L	0.54
B-New	AL-11268-L	0.62
B-Used	AL-11269-L	0.62
C-New	AL-11250-L	0.01
C-Used	AL-11251-L	0.02
D-New	AL-11254-L	0.07
D-Used	AL-11255-L	0.07
E-New	AL-11256-L	*15.8
E-Used	AL-11257-L	*15.7
F-New	AL-11258-L	0.42
F-Used	AL-11259-L	0.51
G-New	AL-11260-L	3.2
G-Used	AL-11261-L	3.5
H-New	AL-11262-L	0.34
H-Used	AL-11263-L	0.34
I-New	AL-11264-L	0.34
I-Used	AL-11265-L	0.38
J-New	AL-11270-L	0.51
J-Used	AL-11271-L	0.38
K-New	AL-11266-L	0.48
K-Used	AL-11267-L	0.43

*Strong Acid Value = 7.1 on sample AL-11256-L and AL-11257-L

TABLE A-4. ANALYTICAL REPORT
SYNTHETIC LUBRICANT ANALYSIS

No. of Particles/100 ml
Particle Sizes in Micrometers

NASA-Lewis Description	SwRI Oil Code	5-15	15-25	25-50	50-100	100	Fibers
A-New	AL-11252-L	17	2	2	4	10	12
A-Used	AL-11253-L	4	1	6	7	11	10
B-New	AL-11268-L	6800	2980	200	40	44	112
B-Used	AL-11269-L	49	51	27	23	16	18
C-New	AL-11250-L	72	36	18	12	10	7
C-Used	AL-11251-L	4	1	2	1	5	9
D-New	AL-11254-L	685	275	35	22	15	20
D-Used	AL-11255-L	200	65	38	24	21	39
E-New	AL-11256-L	120	60	23	25	22	33
E-Used	AL-11257-L	44	7	10	13	12	19
F-New	AL-11258-L	60	16	30	13	7	22
F-Used	AL-11259-L	475	8	2	5	6	52
G-New	AL-11260-L	49	39	45	38	34	78
G-Used	AL-11261-L	4740	10	11	9	6	34
H-New	AL-11262-L	1780	72	45	40	25	32
H-Used	AL-11263-L	1850	118	108	60	52	62
I-New	AL-11264-L	54	23	17	16	4	19
I-Used	AL-11265-L	840	660	450	210	80	120
J-New	AL-11270-L	47	22	10	7	12	18
J-Used	AL-11271-L	36	18	14	8	11	29
K-New	AL-11266-L	185	175	100	70	35	45
K-Used	AL-11267-L	105	48	35	21	20	22

TABLE A-5. ANALYTICAL REPORT
SYNTHETIC LUBRICANT ANALYSIS

NASA-Lewis Description	Oil Code	Elements by XRF (ppm)* Mg	Al	Cl	Fe	Ni	Cu	Pb	Zn(1)	P(2)	S(2)	Ca(2)	Ba(2)	Si	Mn	Limit(3) of Detection (ppm)
A-New	AL-11252-L	0.48	-	2.47	-	-	-	0.21	-	0.18	4.71	-	0.23	-	-	0.11
A-Used	AL-11253-L	-	5.91	1.12	0.51	0.10	0.14	-	0.11	0.17	1.12	-	0.12	-	-	0.09
B-New	AL-11268-L	0.86	-	1.80	-	-	-	-	0.88	0.47	10.40	-	-	0.33	-	0.21
B-Used	AL-11269-L	0.60	4.00	1.90	0.57	-	-	-	0.74	0.27	7.40	-	-	0.90	-	0.23
C-New	AL-11250-L	0.28	-	0.73	0.13	-	-	-	-	0.26	-	-	-	-	-	0.09
C-Used	AL-11251-L	-	2.97	1.04	2.19	0.21	0.12	-	0.15	0.19	0.20	-	-	-	-	0.09
D-New	A. '1254-L	0.27	-	0.90	-	-	-	-	-	0.16	-	-	-	-	-	0.11
D-Used	AL-11255-L	-	12.7	2.08	1.16	0.24	0.19	0.20	0.20	0.71	0.51	-	-	-	-	0.15
E-New	AL-11256-L	0.16	0.19	7.57	0.10	-	-	1.28	7.27	2.15	13.01	0.29	10.16	-	-	0.09
E-Used	AL-11257-L	0.12	1.69	1.61	0.26	-	0.11	-	3.71	0.94	4.29	-	2.43	-	-	0.09
F-New	AL-11258-L	0.31	-	0.45	-	-	-	-	-	0.19	7.08	-	-	-	-	0.10
F-Used	AL-11259-L	5.36	-	2.49	-	-	-	-	-	2.42	51.0	-	-	-	-	0.55
G-New	AL-11260-L	1.31	-	4.91	-	-	-	-	1.51	0.70	5.29	8.69	-	-	-	0.43
G-Used	AL-11261-L	0.39	0.67	1.49	0.22	-	-	-	0.39	-	0.89	2.53	-	-	-	0.13
H-New	AL-11262-L	0.29	-	3.81	0.11	-	-	0.16	-	0.47	0.21	-	-	-	-	0.10
H-Used	AL-11263-L	0.67	4.68	16.68	0.74	-	0.26	-	0.62	2.37	3.20	3.47	-	-	-	0.25
I-New	AL-11264-L	0.33	-	0.56	-	-	-	0.11	-	0.58	-	-	-	-	-	0.10
I-Used	AL-11265-L	0.34	1.18	0.85	0.58	-	-	0.12	0.13	0.46	0.16	-	-	-	-	0.11
J-New	AL-11270-L	0.23	-	0.29	0.07	-	-	-	0.02	0.29	0.06	-	-	-	0.10	0.07
J-Used	AL-11271-L	0.56	-	0.37	0.11	-	0.21	-	0.29	1.11	0.31	-	-	-	-	0.08
K-New	AL-11266-L	0.60	-	9.80	0.28	-	-	-	-	2.51	-	-	-	-	-	0.24
K-Used	AL-11267-L	1.26	0.39	7.30	0.56	-	-	0.65	-	1.86	-	-	-	-	-	0.37

(1)Zn could be due to wear when present with copper, or as an additive when present alone.
(2)P, S, Ca, Ba probably present as additives.
(3)Limit of detection for sample, when - shown, element is less than this value.
*See page __ . Notes on XRF Particulate Wear Metal Analysis.

TABLE A-6. SYNTHETIC LUBRICANT ANALYSIS

NASA-Lewis Description	SwRI Oil Code	Notes on XRF Particulate Metal Analysis
A-New	AL-11252	P, Ba and S present from additive package.
A-Used	AL-11253	Al, Fe, Ni, Cu+Zn present as wear metal particles.
B-New	AL-11268	High S probably from additives.
B-Used	AL-11269	Al, Fe, Zn present as wear metal particles.
C-New	AL-11250	P present as additive.
C-Used	AL-11251	Al, Fe, Ni, Cu, and Zn present as wear metal particles.
D-New	AL-11254	
D-Used	AL-11255	Al, Fe, Ni, Cu, Pb, Zn present as wear metal particles.
E-New	AL-11256	High Zn content and Cl content as well as P, S, Ca, Ba, all from additive package. This additive package more typical of reciprocating piston engine oil. Strong acid probably due to free sulfonic acid from the additives.
E-Used	AL-11257	Al, Fe, Cu present as wear metal particles.
F-New	AL-11258	Oil as received had tarry deposit at bottom of jar which could not be redissolved or suspended in oil.
F-Used	AL-11259	A precipitate of rather large particles at the bottom of the jar could be resuspended, but made filtration difficult and caused a reduction in sample size used for XRF analysis.
G-New	AL-11260	Some plugging of the filter caused a reduction in sample size used in XRF analysis. High S, Ca, Zn probably from additives.
G-Used	AL-11261	Al, Fe present as wear metal particles.
H-New	AL-11262	
H-Used	AL-11263	Al, Fe, Cu, Zn present as wear metal particles.
I-New	AL-11264	
I-Used	AL-11265	Al, Fe, Zn present as wear metal particles.
J-New	AL-11270	
J-Used	AL-11271	Fe, Cu present as wear metal particles.
K-New	AL-11266	
K-Used	AL-11267	Al, Fe, Pb present as wear meatl particles.

TABLE A-7. ANALYTICAL REPORT

SYNTHETIC LUBRICANT ANALYSIS

TOTAL IRON ANALYSIS BY COLORIMETRIC METHOD*

NASA-Lewis Description	SwRI Oil Code	Iron Content (PPM)
A-New	AL-11252-L	1
A-Used	AL-11253-L	4
B-New	AL-11268-L	<1
B-Used	AL-11269-L	<1
C-New	AL-11250-L	1
C-Used	AL-11251-L	6
D-New	AL-11254-L	<1
D-Used	AL-11255-L	1
E-New	AL-11256-L	<1
E-Used	AL-11257-L	1
F-New	AL-11258-L	<1
F-Used	AL-11259-L	2
G-New	AL-11260-L	2
G-Used	AL-11261-L	3
H-New	AL-11262-L	<1
H-Used	AL-11263-L	1
I-New	AL-11264-L	<1
I-Used	AL-11265-L	<1
J-New	AL-11270-L	<1
J-Used	AL-11271-L	<1
K-New	AL-11266-L	<1
K-Used	AL-11267-L	<1

*Technical Report AFWAL-TR-80-4022

TABLE A-8. ANALYTICAL REPORT
SYNTHETIC LUBRICANT ANALYSIS

NASA-Lewis Description	SwRI Oil Code	Specific Heat Measurement @ Listed Temperature					
		82°C		100°C		140°C	
		Cp†	σ	Cp†	σ	Cp†	σ
A-New	AL-11252-L	0.42	0.091	0.42	0.12	0.44	0.14
A-Used	AL-11253-L	0.41	0.094	0.42	0.088	0.41	0.071*
B-New	AL-11268-L	0.50	0.048	0.50	0.051	0.49	0.070
B-Used	AL-11269-L	0.49	0.040	0.48	0.038	0.49	0.059
C-New	AL-11250-L	0.33	0.097	0.32	0.097	0.32	0.091
C-Used	AL-11251-L	0.42	0.026	0.40	0.024	0.40	0.044
D-New	AL-11254-L	0.33	0.071	0.34	0.072	0.34	0.084*
D-Used	AL-11255-L	0.51	0.048	0.52	0.092	0.46	0.14*
E-New	AL-11256-L	0.68	0.11	0.73	0.13	0.76	0.20
E-Used	AL-11257-L	0.60	0.063	0.59	0.069	0.58	0.066
F-New	AL-11258-L	0.53	0.12	0.54	0.13	0.54	0.14
F-Used	AL-11259-L	0.62	0.014	0.62	0.014	0.61	0.013*
G-New	AL-11260-L	0.50	0.091	0.47	0.058	0.42	0.059
G-Used	AL-11261-L	0.53	0.13	0.49	0.12	0.47	0.15
H-New	AL-11262-L	0.37	0.036	0.30	0.037	0.31	0.094
H-Used	AL-11263-L	0.45	0.026	0.35	0.037	0.32	0.040*
I-New	AL-11264-L	0.53	0.060	0.47	0.039	0.44	0.075*
I-Used	AL-11265-L	0.48	0.087	0.40	0.085	0.40	0.10
J-New	AL-11270-L	0.47	0.031	0.48	0.030	0.49	0.030
J-Used	AL-11271-L	0.34	0.025	0.34	0.028	0.34	0.029
K-New	AL-11266-L	0.44	0.073	0.38	0.076	0.34	0.075
K-Used	AL-11267-L	0.36	0.098	0.27	0.11	0.27	0.11*

*For calculation of Cp and σ (standard deviation) one value, inordinately different from the others, was discarded. Thus, four values rather than five were used to determine these data.

†Cp= (mcal/mg deg C)

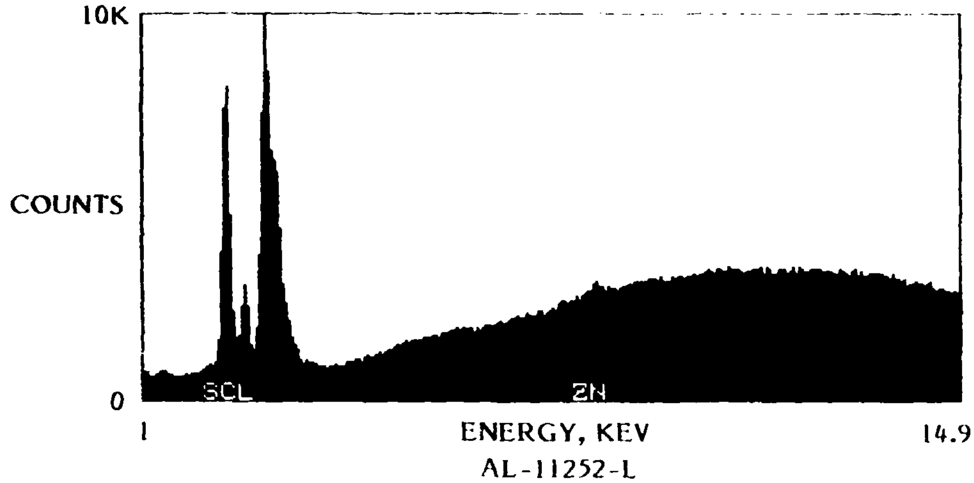


FIGURE A-1. NASA-A

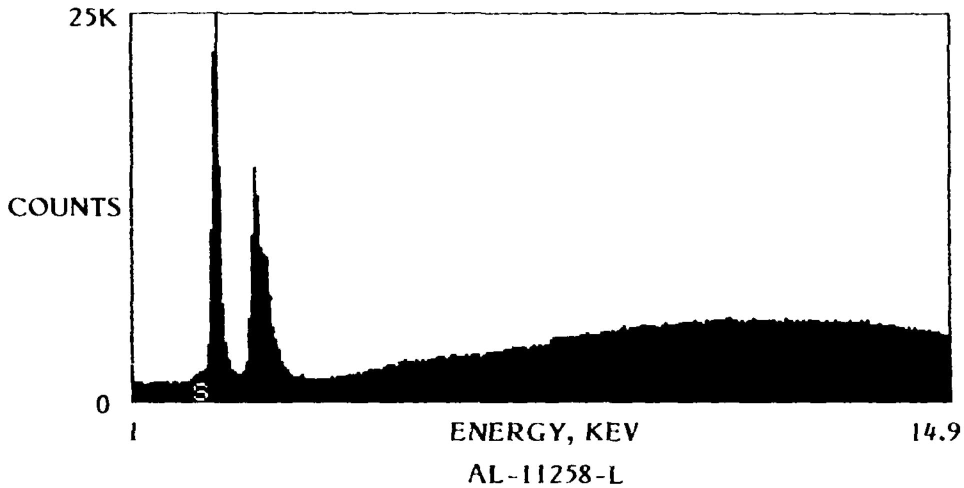


FIGURE A-2. NASA-B

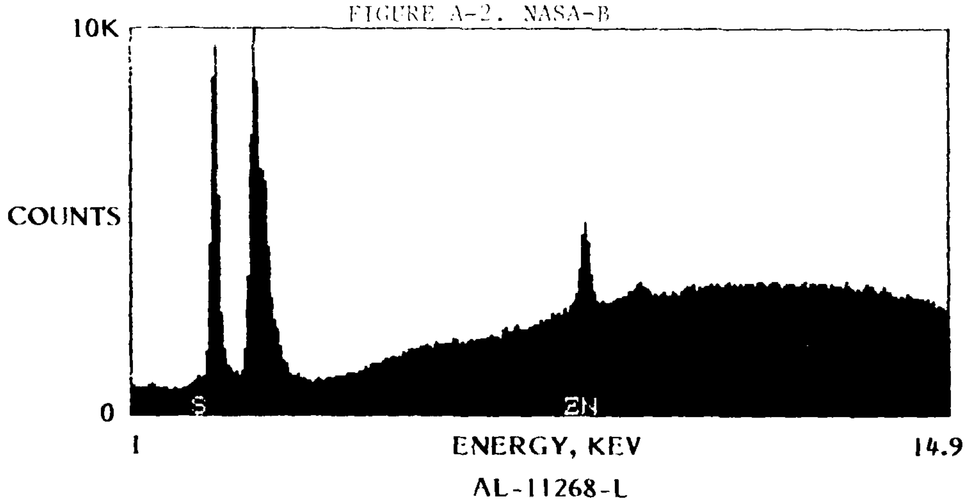
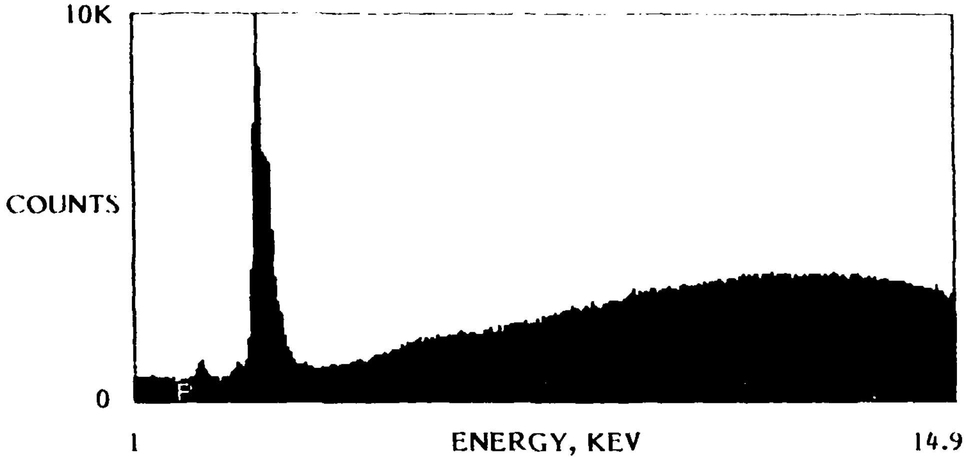
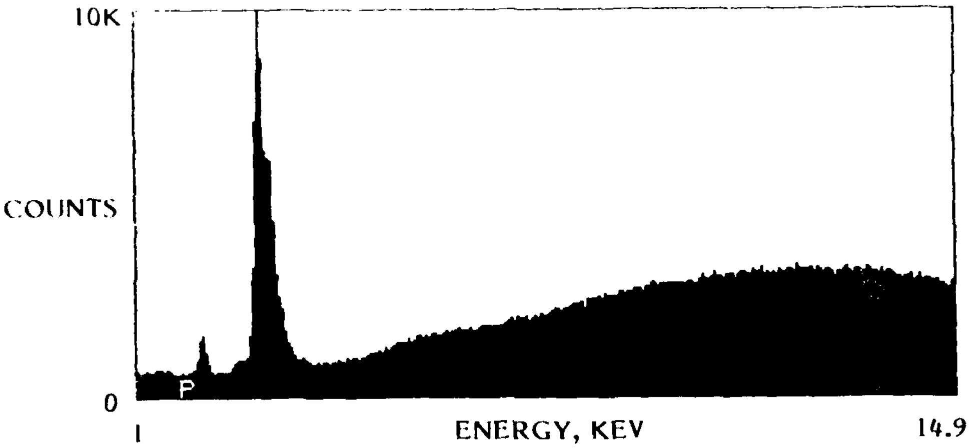


FIGURE A-3. NASA-C



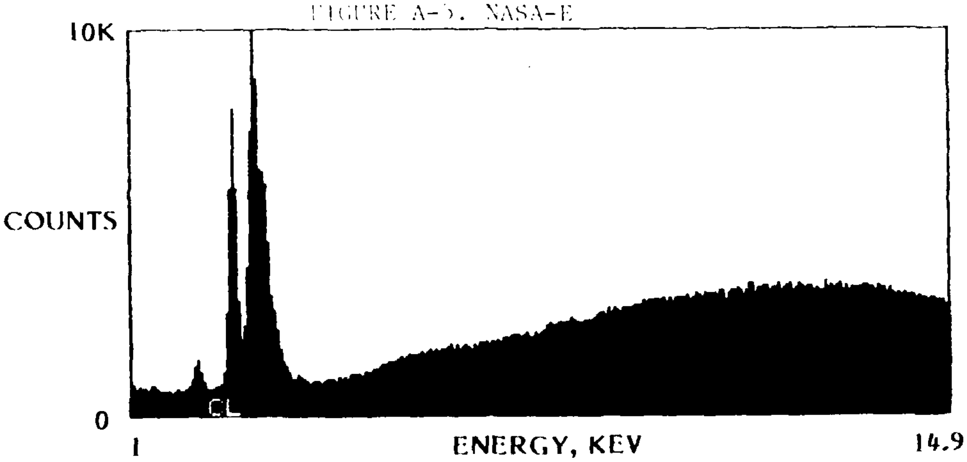
AL-11250-L

FIGURE A-4. NASA-D



AL-11254-L

FIGURE A-5. NASA-E



AL-11262-L

FIGURE A-6. NASA-F

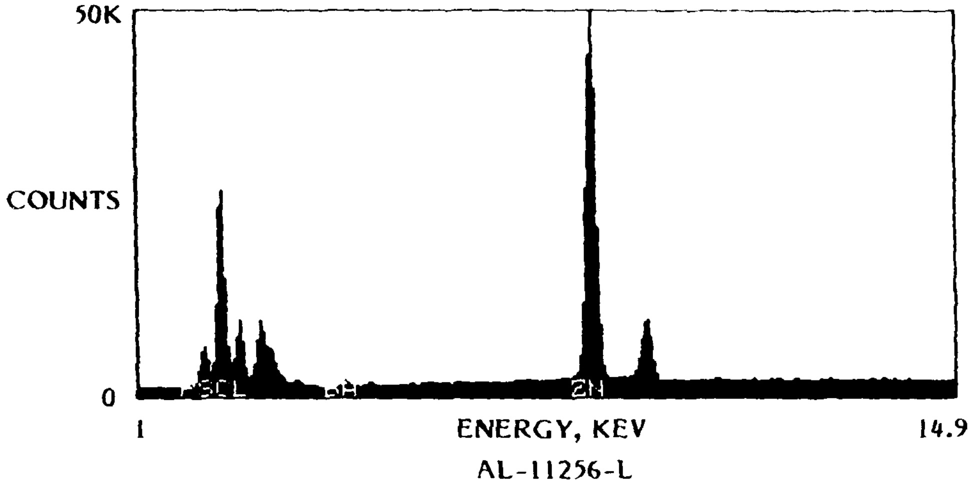


FIGURE A-7. NASA-G

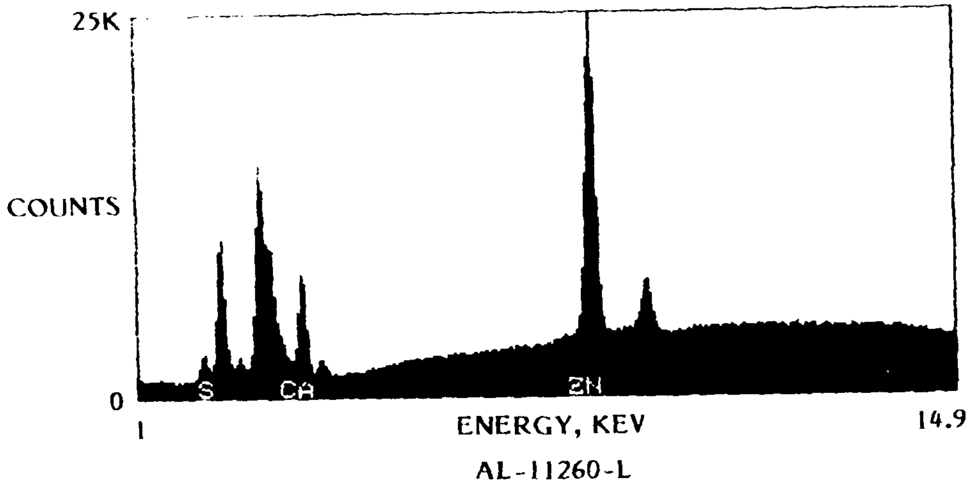
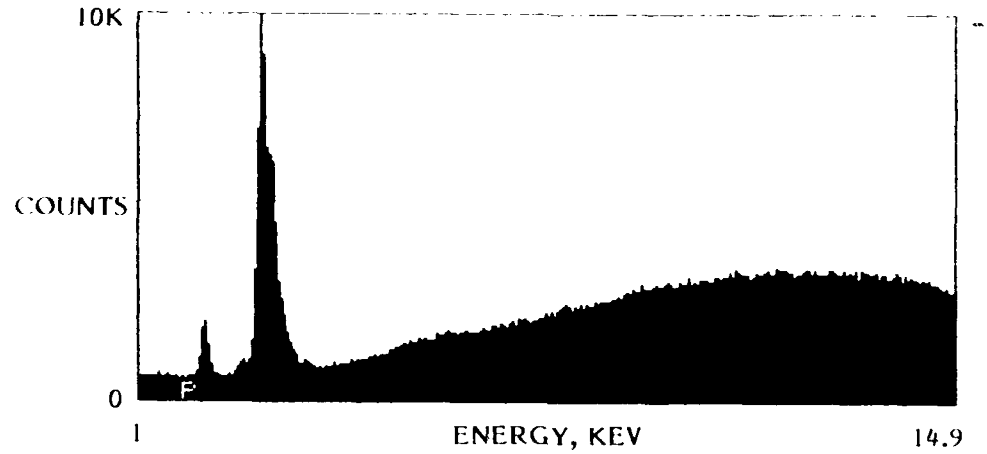
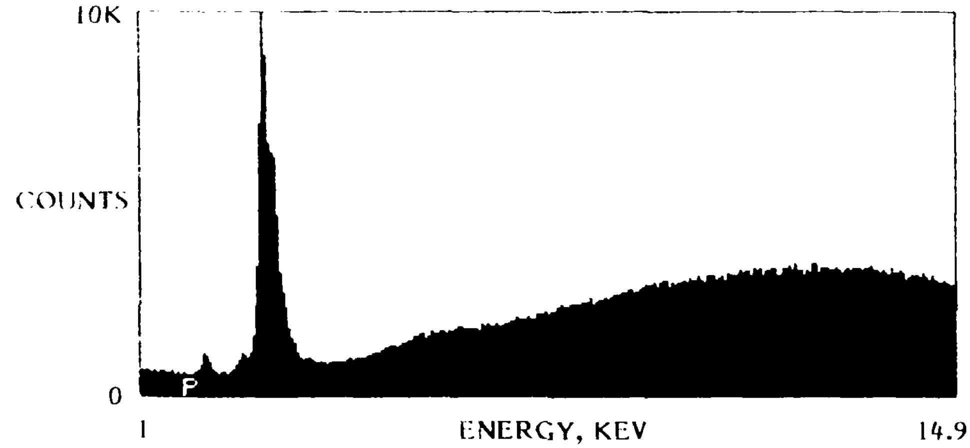


FIGURE A-8. NASA-H



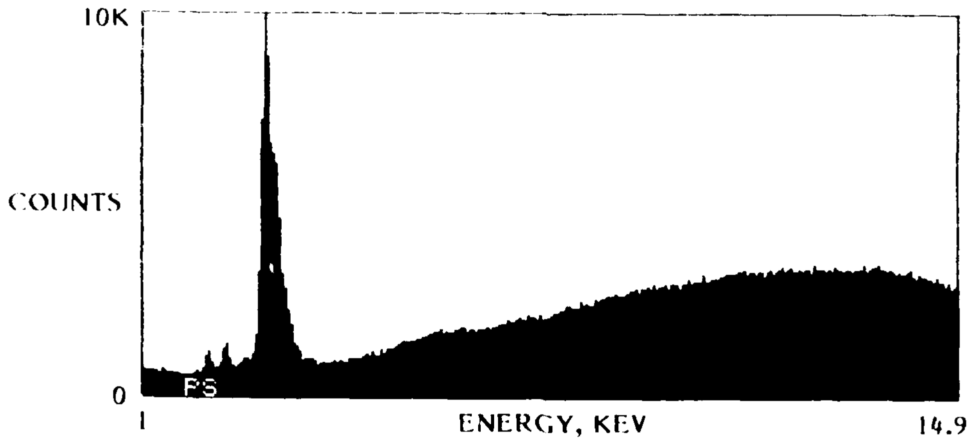
AL-11264-L

FIGURE A-9. NASA-I



AL-11266-L

FIGURE A-10. NASA-J



AL-11270-L

FIGURE A-11. NASA-K

APPENDIX B

FRICTION AND WEAR TEST DATA

TABLE B-1. LFW-1 FRICTION AND WEAR TEST RESULTS

Test Lubricant NASA Code	SwRI Code	Coefficient Friction After Cycle 400	800	1200	9000	10,000	Avg Coeff of Friction	Mean Coeff of Friction	Avg Wear Scar Width, mm	Mean Wear Scar Width, mm	Weight Loss, mg Block	Ring	Total	Mean
A	AL-11252-L	0.047	0.047	0.059	0.065	0.059	0.055	0.053	1.12	1.07	0.5	1.4	1.9	2.0
A	AL-11252-L	0.047	0.044	0.053	0.053	0.053	0.050		1.02		0.4	1.7	2.1	
B	AL-11268-L	0.026	0.024	0.024	0.029	0.029	0.026	0.027	1.02	0.91	0.5	1.6	2.1	2.0
B	AL-11268-L	0.026	0.026	0.026	0.029	0.029	0.027		0.79		0.8	1.1	1.9	
C	AL-11250-L	0.032	0.026	0.026	0.021	0.021	0.025	0.024	1.14	1.12	0.7	1.7	2.4	3.3
C	AL-11250-L	0.026	0.021	0.018	0.029	0.018	0.022		1.09		2.7	1.4	4.1	
D	AL-11254-L	0.026	0.023	0.021	0.015	0.015	0.020	0.017	1.19	1.12	1.4	1.5	2.9	2.4
D	AL-11254-L	0.018	0.015	0.015	0.012	0.012	0.014		1.04		0.2	1.7	1.9	
E	AL-11256-L	0.029	0.029	0.026	0.026	0.026	0.027	0.035	1.04	0.95	1.3	0.9	2.2	2.0
E	AL-11256-L	0.044	0.041	0.041	0.041	0.041	0.042		0.86		0.2	1.6	1.8	
F	AL-11258-L	0.044	0.044	0.041	0.056	0.059	0.049	0.034	1.02	1.02	1.4	1.7	3.1	2.5
F	AL-11258-L	0.024	0.021	0.018	0.015	0.015	0.019		1.02		0.1	1.7	1.8	
G	AL-11260-L	0.047	0.047	0.053	0.071	0.071	0.058	0.043	1.22	1.10	1.1	1.3	2.4	2.0
G	AL-11260-L	0.026	0.026	0.026	0.032	0.032	0.028		0.97		0.2	1.4	1.6	
H	AL-11262-L	0.021	0.021	0.015	0.018	0.021	0.019	0.022	0.99	1.11	1.4	1.8	3.2	2.8
H	AL-11262-L	0.029	0.026	0.024	0.024	0.021	0.025		1.22		0.2	2.1	2.3	
I	AL-11264-L	0.035	0.029	0.026	0.041	0.041	0.034	0.034	1.19	1.21	1.1	1.3	2.4	2.6
I	AL-11264-L	0.038	0.038	0.038	0.029	0.029	0.034		1.22		0.3	2.5	2.8	
J	AL-11270-L	0.047	0.047	0.041	0.026	0.026	0.037	0.031	1.07	1.06	0.3	1.0	1.3	1.9
J	AL-11270-L	0.029	0.029	0.029	0.018	0.018	0.025		1.04		1.0	1.5	2.5	
K	AL-11266-L	0.024	0.021	0.018	0.015	0.015	0.019	0.022	1.12	1.21	0.4	1.0	1.4	1.5
K	AL-11266-L	0.035	0.029	0.026	0.018	0.018	0.025		1.30		0.1	1.4	1.5	

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APPENDIX C

HIGH-PRESSURE VISCOSITY TEST DATA

SWRI/NASA

AL-11252-L

06-11-82

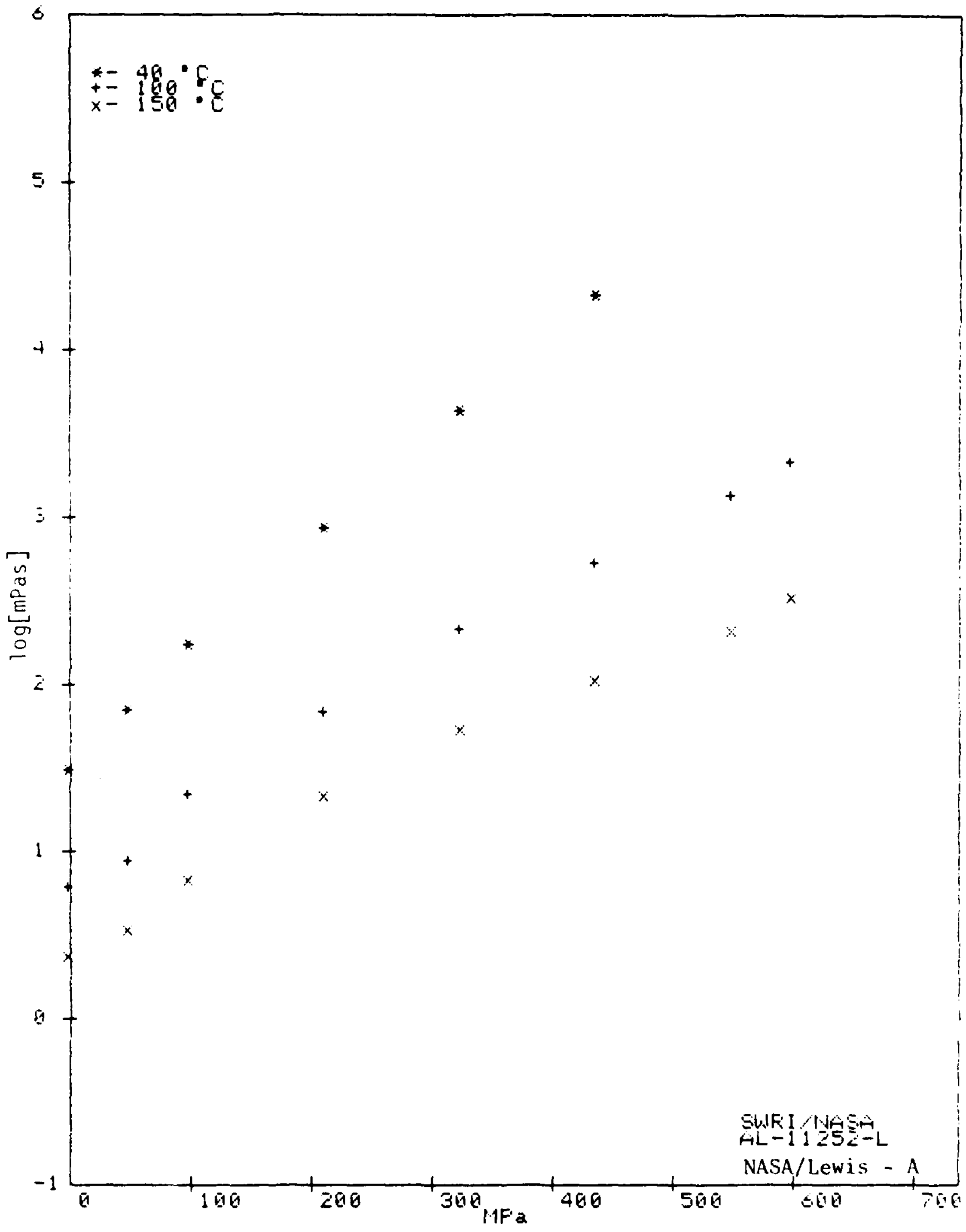
NASA/LEWIS - A

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	32.10
	6.76	46.62	72.10
	14.85	102.40	184.00
	31.03	213.94	986.00
	47.21	325.49	5460.00
	63.39	437.03	27000.00
	79.57	548.56	too high...
100	(1 atm)	0.10	5.94
	6.76	46.62	10.80
	14.85	102.40	22.70
	31.03	213.94	77.60
	47.21	325.49	231.00
	63.39	437.03	671.00
	79.57	548.58	171[illegible].00
	87.66	604.35	26[illegible].00
150	(1 atm)	0.10	2.56
	6.76	46.62	4.24
	14.85	102.40	[illegible]
	31.03	213.94	[illegible]
	47.21	325.49	[illegible]8.70
	63.39	437.03	129.00
	79.57	548.58	273.00
	87.66	604.35	392.00

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	18.30	13.94
100.0	12.87	10.73
150.0	11.20	9.33



*- 40 °C
+- 100 °C
x- 150 °C
log[mPas]
MPa
SWRI/NASA
AL-11252-L
NASA/Lewis - A
6
5
4
3
2
1
0
-1
0
100
200
300
400
500
600
700

SWRI/NASA

AL-11268-L

10-11-82

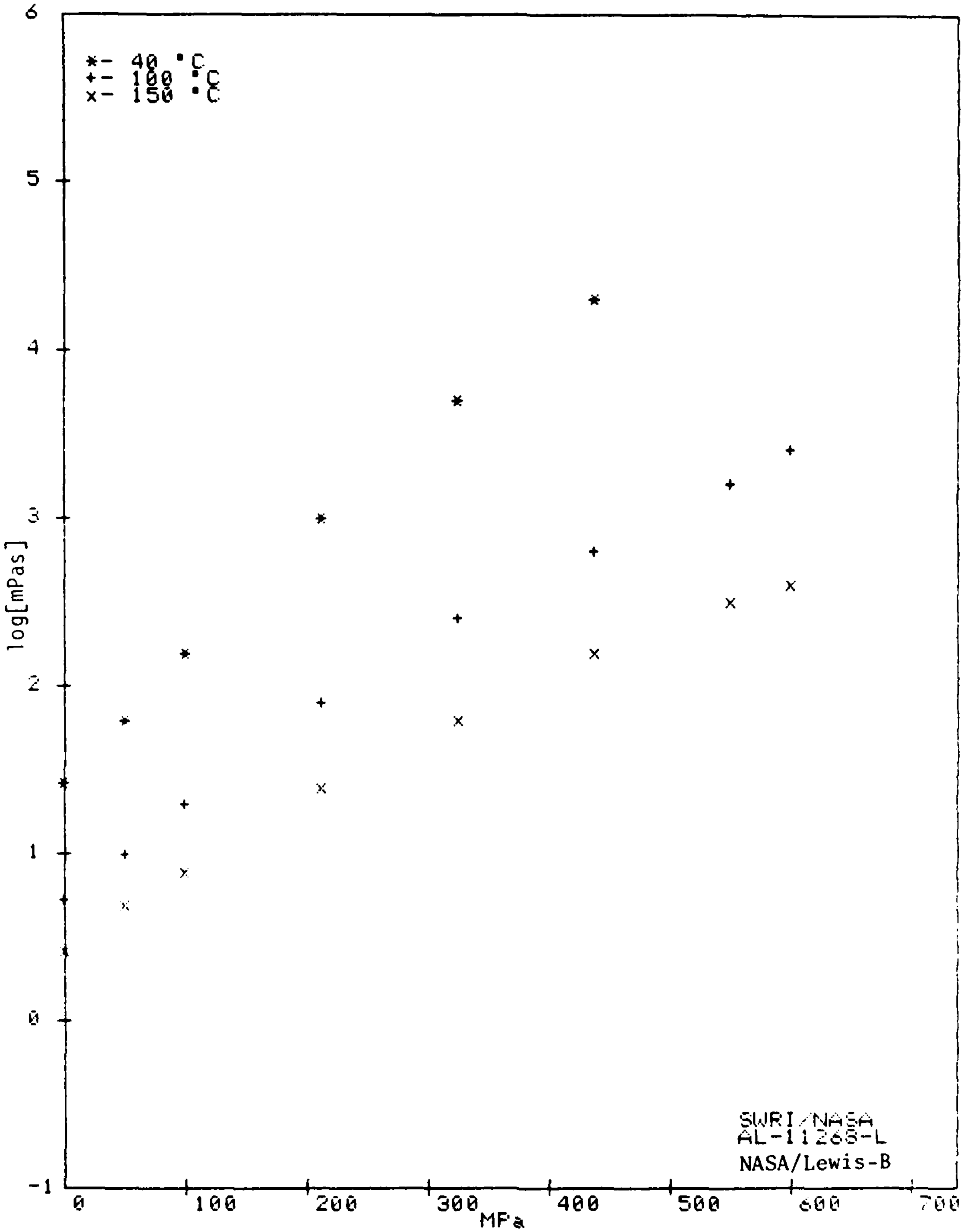
NASA/Lewis - B

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	27.50
	6.76	46.62	58.00
	14.85	102.40	149.00
	31.03	213.94	903.00
	47.21	325.49	4580.00
	63.39	437.03	21100.00
	79.57	548.58	too high...
100	(1 atm)	0.10	5.56
	6.76	46.62	10.20
	14.85	102.40	21.30
	31.03	213.94	76.40
	47.21	325.49	233.00
	63.39	437.03	643.00
	79.57	548.58	1660.00
	87.66	604.35	2560.00
150	(1 atm)	0.10	2.67
	6.76	46.62	4.50
	14.85	102.40	8.65
	31.03	213.94	25.50
	47.21	325.49	63.80
	63.39	437.03	144.00
	79.57	548.58	285.00
	87.66	604.35	389.00

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	16.22	13.58
100.0	13.10	10.84
150.0	11.10	9.42



*- 40 °C
+- 100 °C
x- 150 °C
SWRI/NASA
AL-11268-L
NASA/Lewis-B
log[mPas]
MPa
6
5
4
3
2
1
0
-1
0
100
200
300
400
500
600
700

SWRI/NASA

AL-11250-L

18-08-82

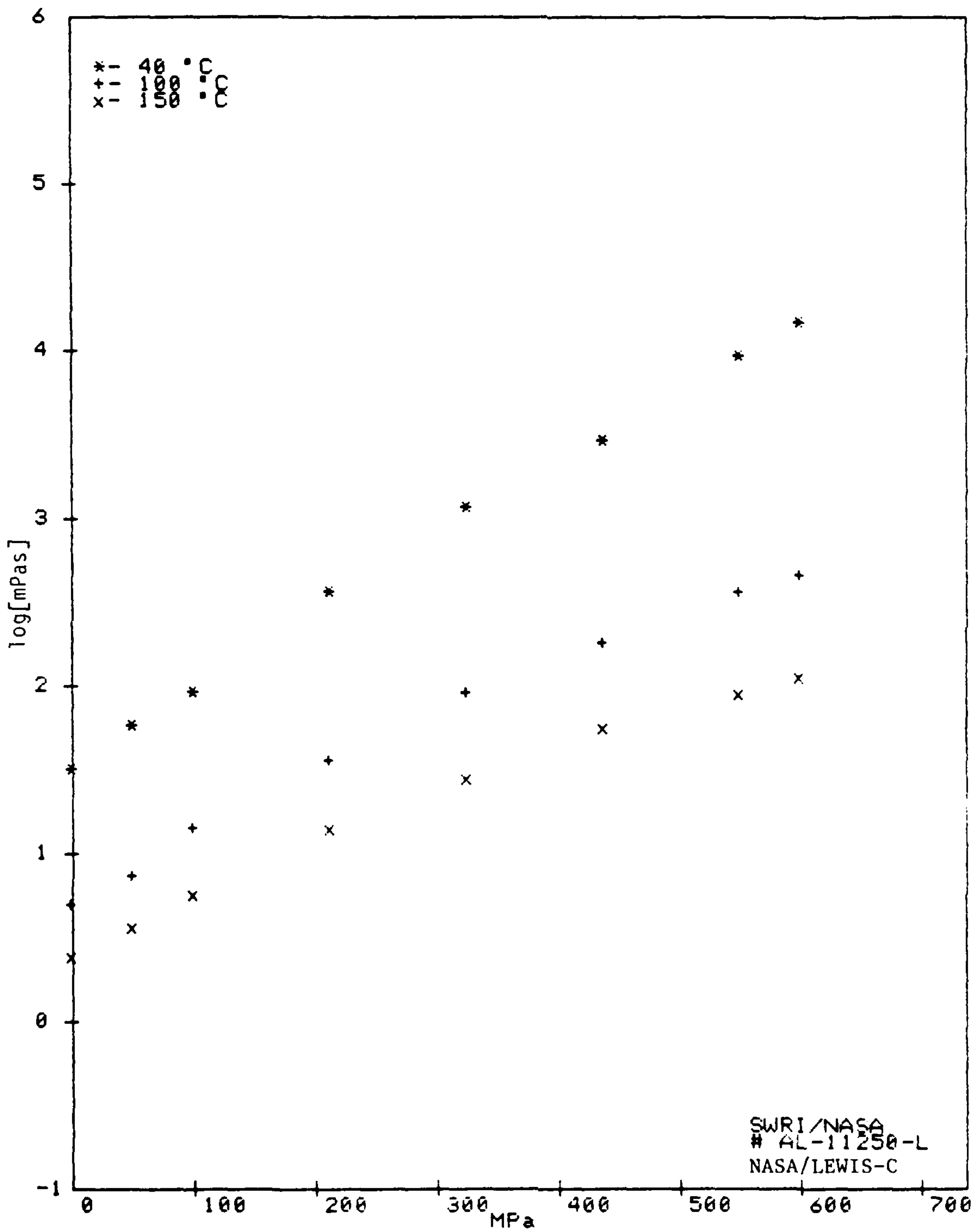
NASA/Lewis - C

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	30.10
	6.76	46.62	56.50
	14.85	102.40	111.00
	31.03	213.94	368.00
	47.21	325.49	1190.00
	63.39	437.03	3180.00
	79.57	548.58	9070.00
	87.66	604.35	16000.00
100	(1 atm)	0.10	4.89
	6.76	46.62	8.53
	14.85	102.40	15.30
	31.03	213.94	39.30
	47.21	325.49	101.00
	63.39	437.03	217.00
	79.57	548.58	422.00
	87.66	604.35	559.00
150	(1 atm)	0.10	2.44
	6.76	46.62	4.05
	14.85	102.40	6.44
	31.03	213.94	15.30
	47.21	325.49	32.60
	63.39	437.03	57.10
	79.57	548.58	93.50
	87.66	604.35	124.00

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	12.58	11.56
100.0	11.36	9.85
150.0	9.53	8.41


*- 40 °C
+- 100 °C
x- 150 °C
log[mPas]
6
5
4
3
2
1
0
-1
0
100
200
300
400
500
600
700
MPa
SWRI/NASA
AL-11250-L
NASA/LEWIS-C

SWRI/NASA

AL-11254-L

20-08-82

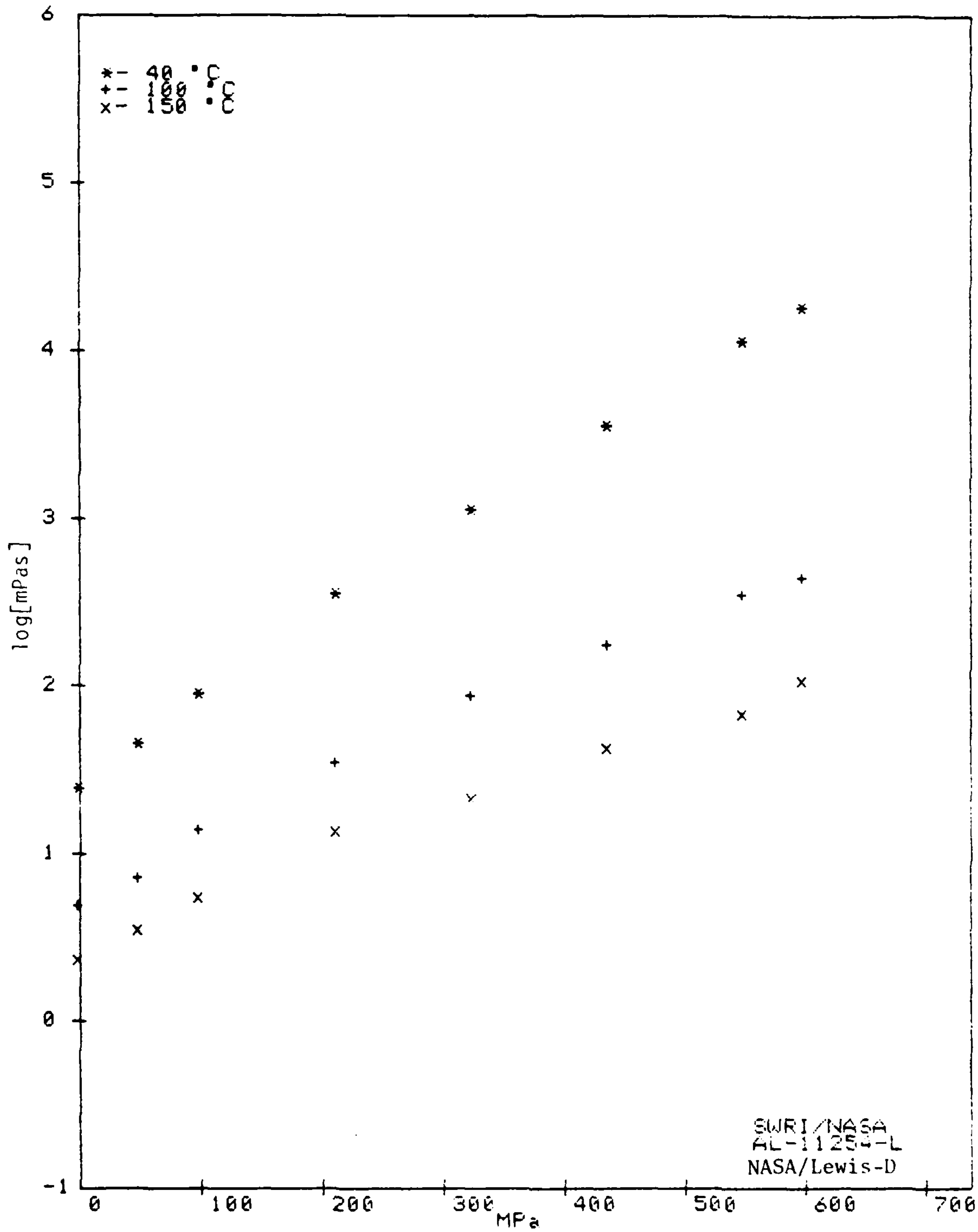
NASA/Lewis - D

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	26.60
	6.76	46.62	50.00
	14.85	102.40	109.00
	31.03	213.94	431.00
	47.21	325.49	1370.00
	63.39	437.03	4270.00
	79.57	548.58	12200.00
	87.66	604.35	21400.00
100	(1 atm)	0.10	4.80
	6.76	46.62	8.23
	14.85	102.40	14.60
	31.03	213.94	40.00
	47.21	325.49	97.80
	63.39	437.03	203.00
	79.57	548.58	429.00
	87.66	604.35	552.00
150	(1 atm)	0.10	2.28
	6.76	46.62	3.56
	14.85	102.40	6.27
	31.03	213.94	14.60
	47.21	325.49	28.00
	63.39	437.03	50.50
	79.57	548.58	88.70
	87.66	604.35	117.00

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	14.04	12.42
100.0	12.52	9.72
150.0	10.00	8.23


*- 40 °C
+- 100 °C
x- 150 °C
6
5
4
3
2
1
0
-1
log[mPas]
0
100
200
300
400
500
600
700
MPa
SWRI/NASA
AL-11254-L
NASA/Lewis-D

SWRI/NASA

AL-11256-L

20-08-82

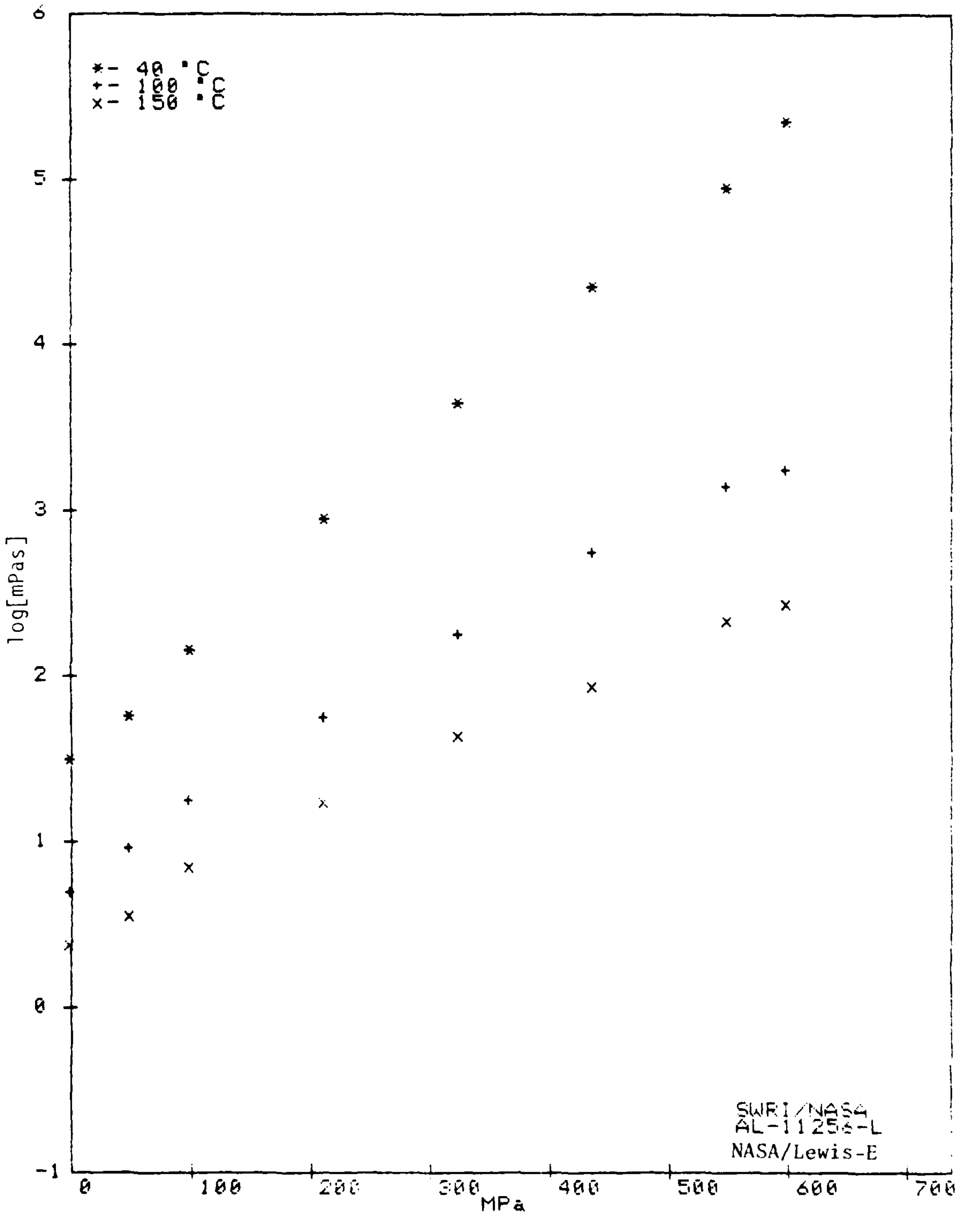
NASA/Lewis - E

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	29.40
	6.76	46.62	65.00
	14.85	102.40	176.00
	31.03	213.94	1010.00
	47.21	325.49	5230.00
	63.39	437.03	22500.00
	79.57	548.58	97200.00
	87.66	604.35	%243000.00
100	(1 atm)	0.10	5.33
	6.76	46.62	9.60
	14.85	102.40	19.40
	31.03	213.94	68.00
	47.21	325.49	214.00
	63.39	437.03	574.00
	79.57	548.58	1430.00
	87.66	604.35	2190.00
150	(1 atm)	0.10	2.39
	6.76	46.62	3.97
	14.85	102.40	7.27
	31.03	213.94	20.30
	47.21	325.49	49.90
	63.39	437.03	110.00
	79.57	548.58	233.00
	87.66	604.35	306.00

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	15.70	15.39
100.0	11.60	11.53
150.0	10.70	9.63



*- 40 °C
+- 100 °C
x- 150 °C
log[mPas]
MPa
SWRI/NASA
AL-11256-L
NASA/Lewis-E
6
5
4
3
2
1
0
-1
0
100
200
300
400
500
600
700

SWRI/NASA

AL-11258-L

23-08-82

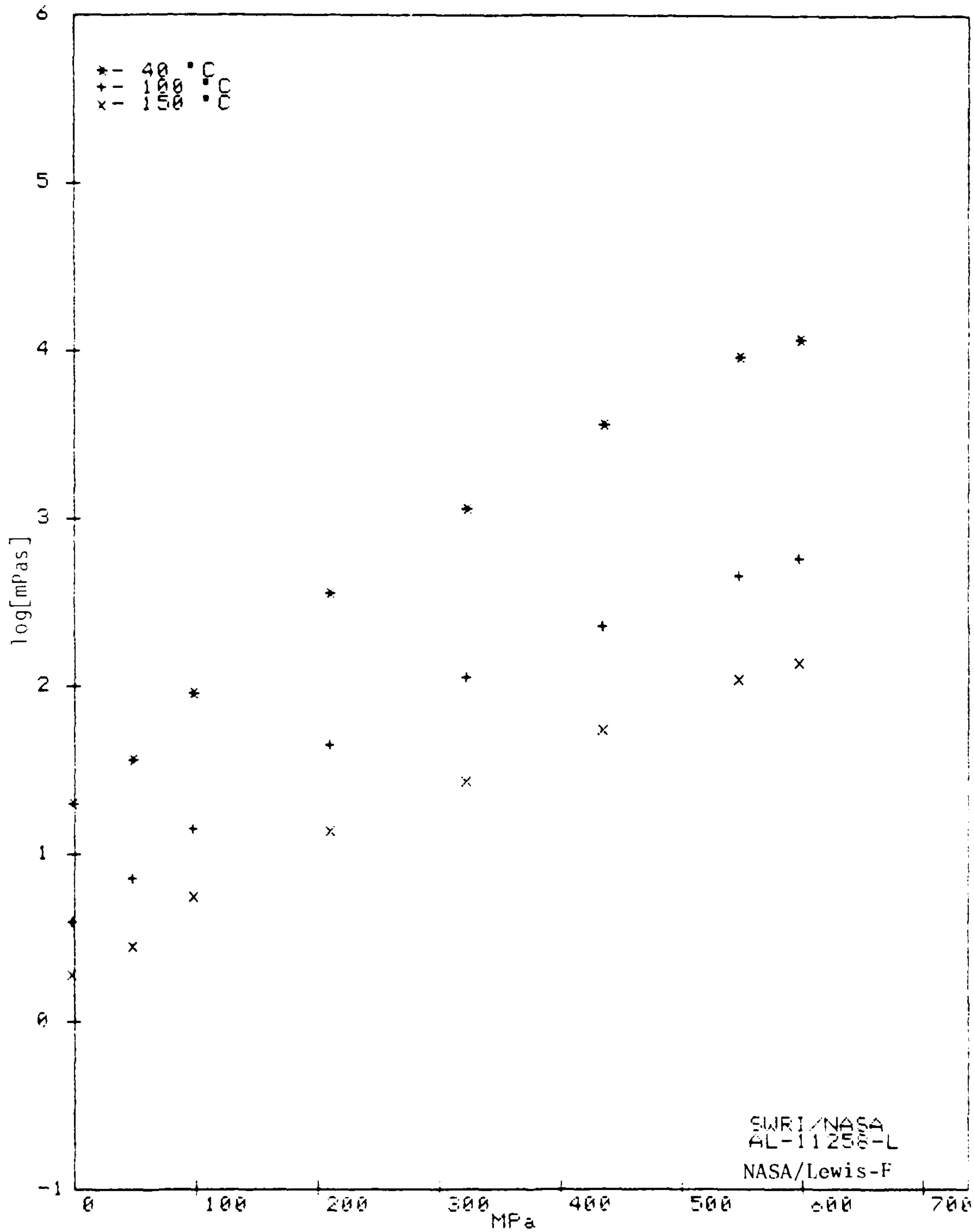
NASA/Lewis - F

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	21.40
	6.76	46.62	43.60
	14.85	102.40	98.40
	31.03	213.94	415.00
	47.21	325.49	1350.00
	63.39	437.03	4060.00
	79.57	548.58	10100.00
	87.66	604.35	13600.00
100	(1 atm)	0.10	4.23
	6.76	46.62	7.80
	14.85	102.40	15.20
	31.03	213.94	45.50
	47.21	325.49	113.00
	63.39	437.03	249.00
	79.57	548.58	512.00
	87.66	604.35	701.00
150	(1 atm)	0.10	1.99
	6.76	46.62	3.31
	14.85	102.40	5.67
	31.03	213.94	15.10
	47.21	325.49	33.10
	63.39	437.03	63.30
	79.57	548.58	115.00
	87.66	604.35	143.00

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	15.00	13.22
100.0	12.45	10.85
150.0	10.76	9.18


*- 40 °C
+- 100 °C
x- 150 °C
SWRI/NASA
AL-11258-L
NASA/Lewis-F
log[mPas]
MPa
6
5
4
3
2
1
0
-1
0
100
200
300
400
500
600
700

SWRI/NASA

AL-11260-L

03-11-82

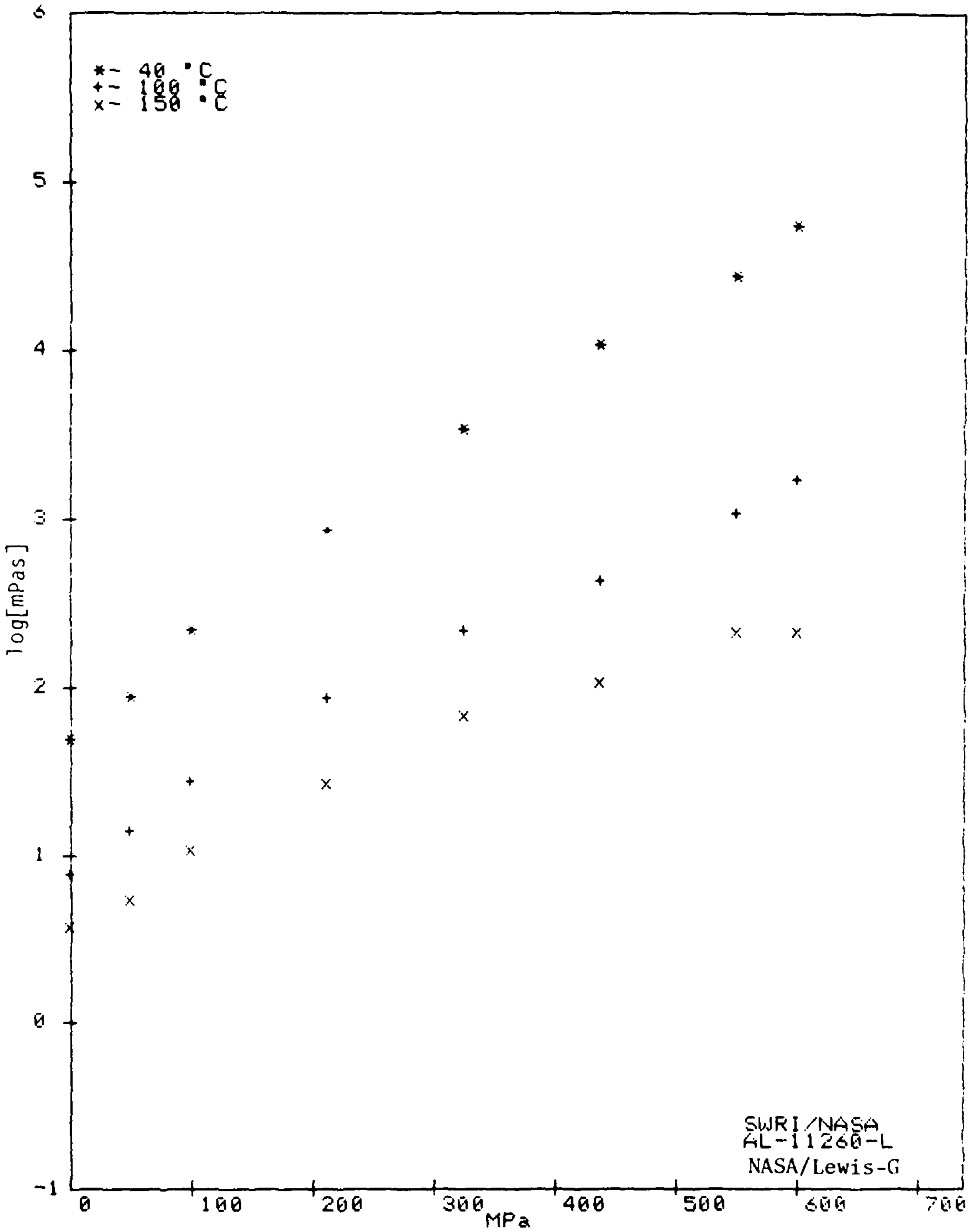
NASA/Lewis - G

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	48.60
	6.76	46.62	103.00
	14.85	102.40	229.00
	31.03	213.94	976.00
	47.21	325.49	4020.00
	63.39	437.03	11600.00
	79.57	548.58	34100.00
	87.66	604.35	70300.00
100	(1 atm)	0.10	8.34
	6.76	46.62	15.40
	14.85	102.40	30.00
	31.03	213.94	103.00
	47.21	325.49	232.00
	63.39	437.03	560.00
	79.57	548.58	1280.00
	87.66	604.35	1830.00
150	(1 atm)	0.10	3.69
	6.76	46.62	6.40
	14.85	102.40	12.30
	31.03	213.94	32.10
	47.21	325.49	75.40
	63.39	437.03	137.00
	79.57	548.58	229.00
	87.66	604.35	254.00

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	14.40	12.58
100.0	12.12	10.34
150.0	10.86	9.24


*- 40 °C
+- 100 °C
x- 150 °C
log[mPas]
6
5
4
3
2
1
0
-1
0
100
200
300
400
500
600
700
MPa
SWRI/NASA
AL-11260-L
NASA/Lewis-G

SWRI/NASA

AL-11262-L

23-08-82

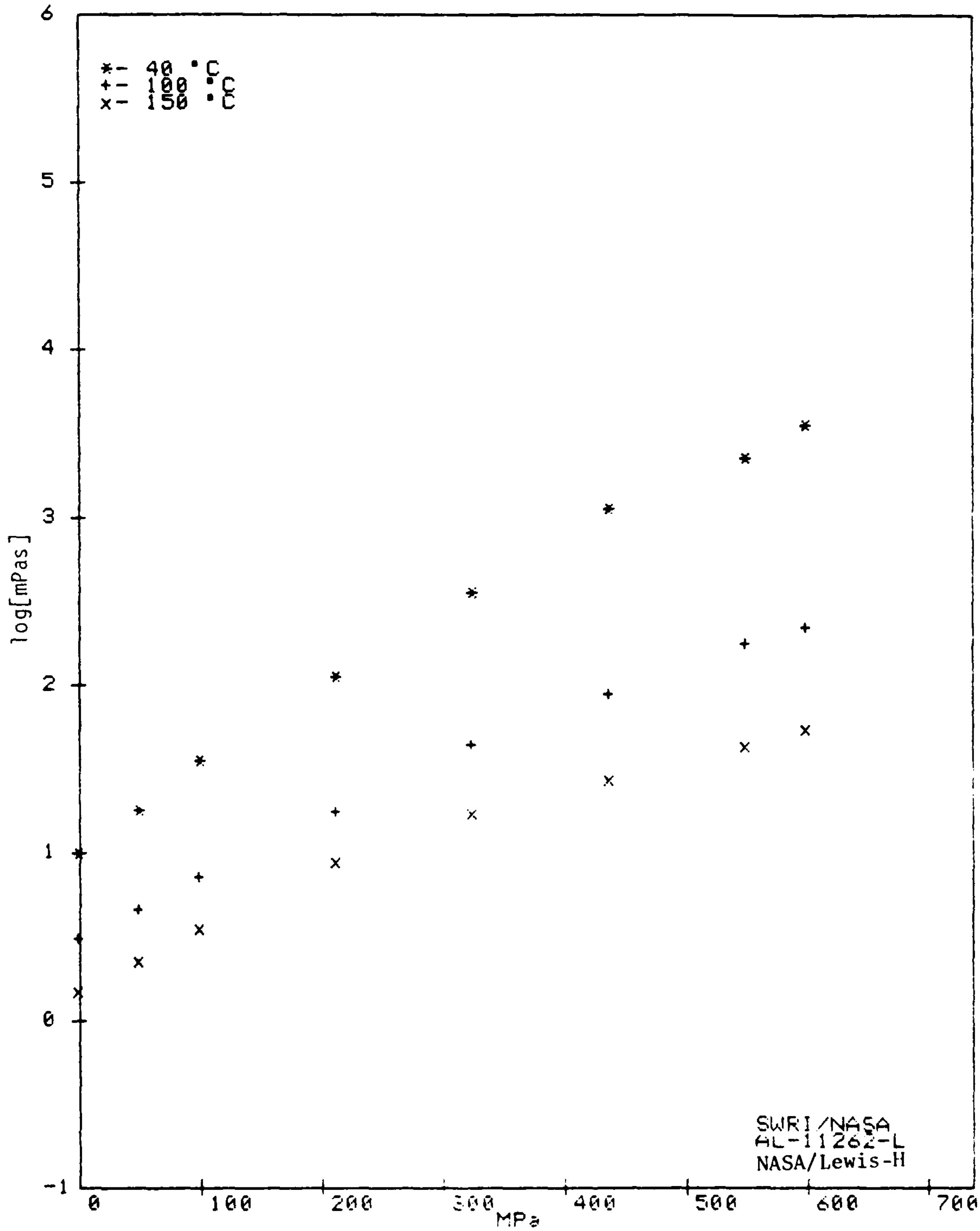
NASA/Lewis - H

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	11.00
	6.76	46.62	20.00
	14.85	102.40	41.40
	31.03	213.94	132.00
	47.21	325.49	394.00
	63.39	437.03	1140.00
	79.57	548.58	2720.00
	87.66	604.35	4270.00
100	(1 atm)	0.10	2.97
	6.76	46.62	4.79
	14.85	102.40	8.29
	31.03	213.94	20.30
	47.21	325.49	48.70
	63.39	437.03	99.70
	79.57	548.58	186.00
	87.66	604.35	246.00
150	(1 atm)	0.10	1.75
	6.76	46.62	2.55
	14.85	102.40	4.20
	31.03	213.94	9.39
	47.21	325.49	19.10
	63.39	437.03	34.30
	79.57	548.58	52.80
	87.66	604.35	68.90

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	12.58	11.45
100.0	9.93	8.79
150.0	8.53	7.47


*- 40 °C
+- 100 °C
x- 150 °C
6
5
4
3
2
1
0
-1
log[mPas]
0
100
200
300
400
500
600
700
MPa
SWRI/NASA
AL-11262-L
NASA/Lewis-H

SWRI/NASA

AL-11264-L

24-08-82

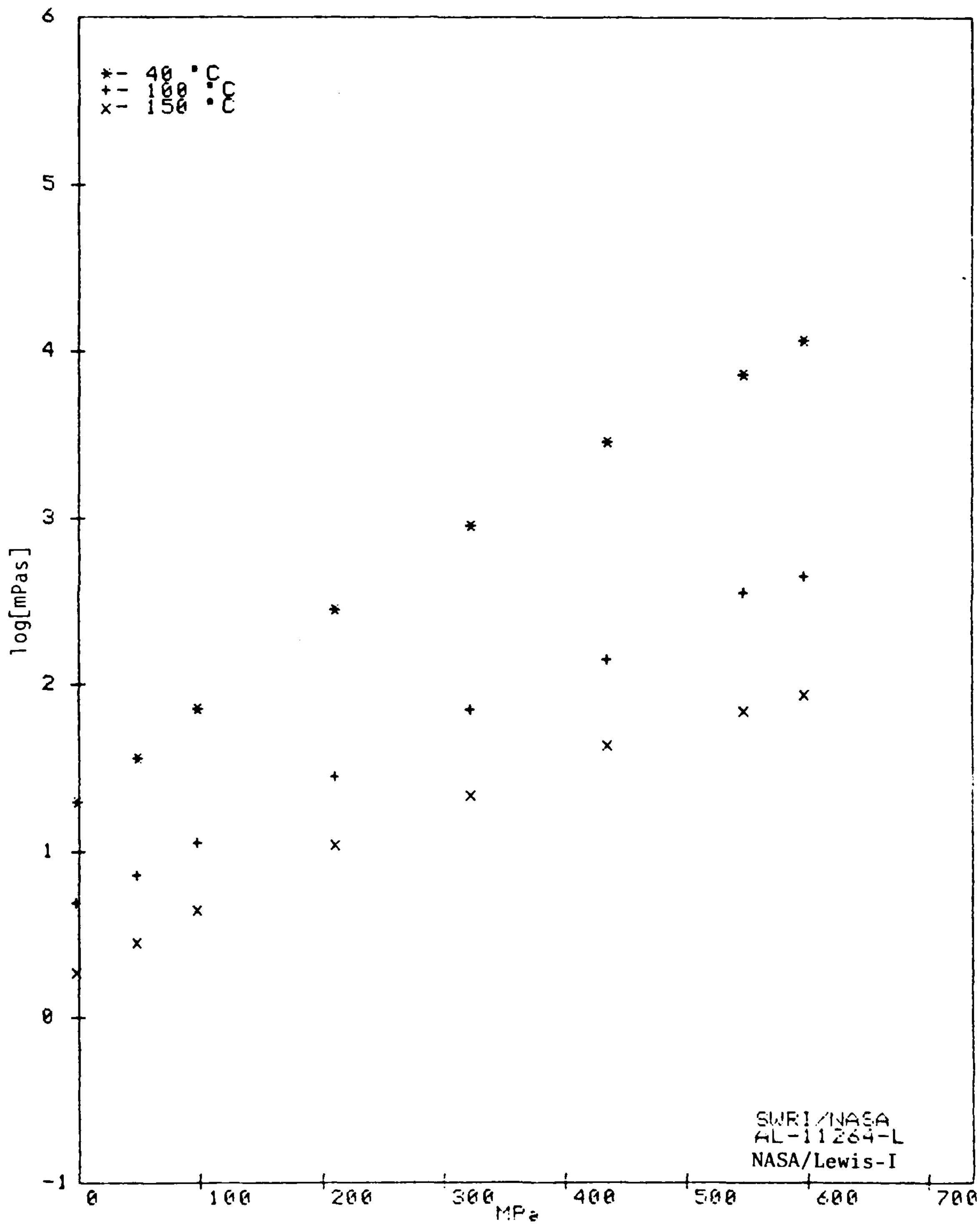
NASA/Lewis - I

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	20.60
	6.76	46.62	38.40
	14.85	102.40	79.90
	31.03	213.94	298.00
	47.21	325.49	1010.00
	63.39	437.03	2910.00
	79.57	548.58	7740.00
	87.66	604.35	12300.00
100	(1 atm)	0.10	4.63
	6.76	46.62	7.28
	14.85	102.40	12.90
	31.03	213.94	34.90
	47.21	325.49	83.00
	63.39	437.03	177.00
	79.57	548.58	364.00
	87.66	604.35	487.00
150	(1 atm)	0.10	2.22
	6.76	46.62	3.47
	14.85	102.40	5.44
	31.03	213.94	13.00
	47.21	325.49	26.50
	63.39	437.03	46.60
	79.57	548.58	81.10
	87.66	604.35	102.00

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	13.16	11.94
100.0	9.52	8.95
150.0	8.86	7.88


*- 40 °C
+- 100 °C
x- 150 °C
log[mPas]
MPa
SWRI/NASA
AL-11264-L
NASA/Lewis-I
6
5
4
3
2
1
0
-1
0
100
200
300
400
500
600
700

SWRI/NASA

AL-11270-L

24-08-82

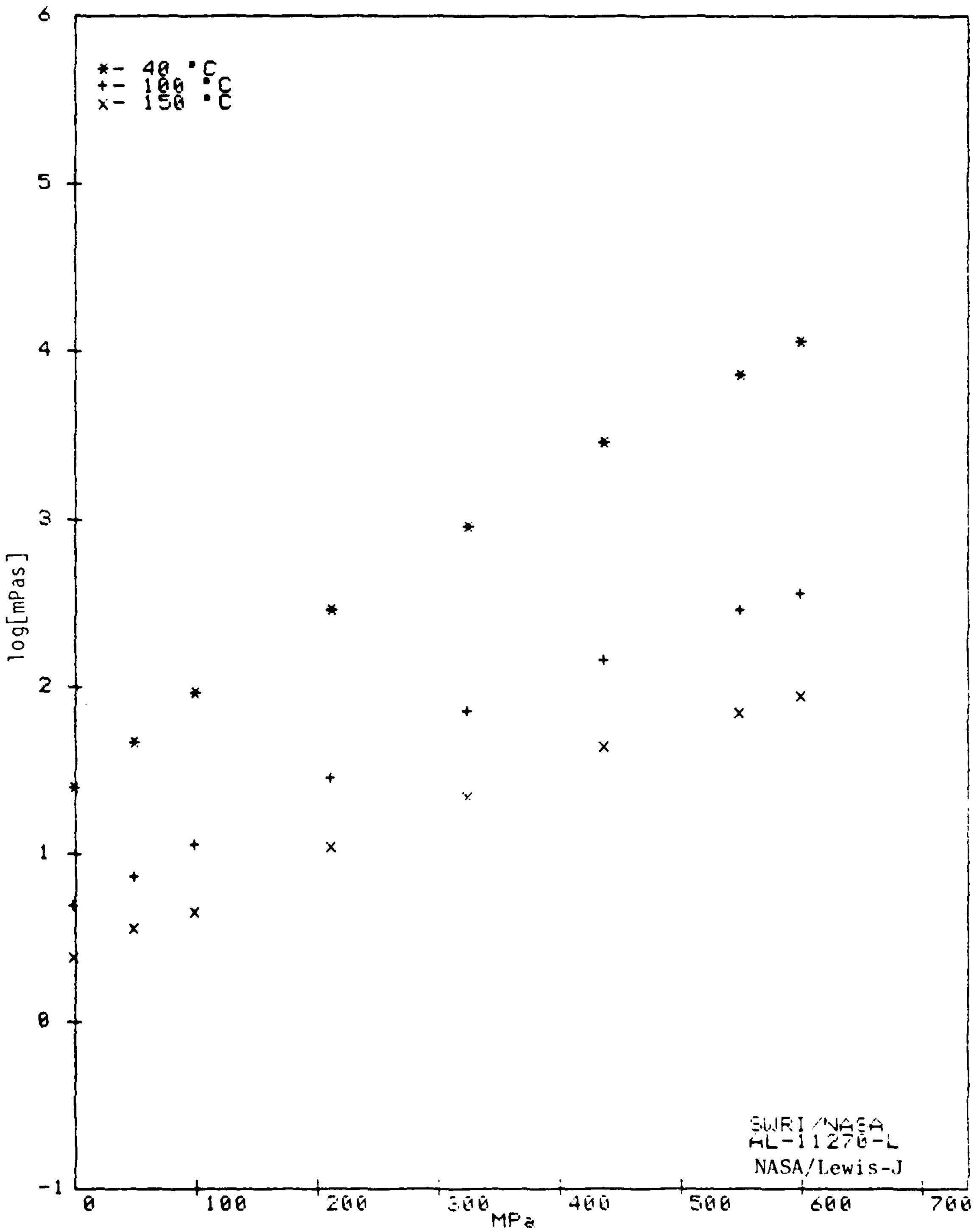
NASA/Lewis - J

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	24.10
	6.76	46.62	44.90
	14.85	102.40	92.60
	31.03	213.94	330.00
	47.21	325.49	1120.00
	63.39	437.03	3150.00
	79.57	548.58	8330.00
	87.66	604.35	13300.00
100	(1 atm)	0.10	4.57
	6.76	46.62	7.12
	14.85	102.40	12.80
	31.03	213.94	35.40
	47.21	325.49	76.80
	63.39	437.03	161.00
	79.57	548.58	312.00
	87.66	604.35	416.00
150	(1 atm)	0.10	2.27
	6.76	46.62	3.58
	14.85	102.40	5.57
	31.03	213.94	13.00
	47.21	325.49	25.90
	63.39	437.03	46.80
	79.57	548.58	77.40
	87.66	604.35	96.20

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	12.80	11.94
100.0	9.80	8.95
150.0	8.37	7.85



*- 40 °C
+- 100 °C
x- 150 °C
6
5
4
3
2
1
0
-1
log[mPas]
0
100
200
300
400
500
600
700
MPa
SWRI/NASA
AL-11270-L
NASA/Lewis-J

SWRI/NASA

AL-11266-L

06/11/82

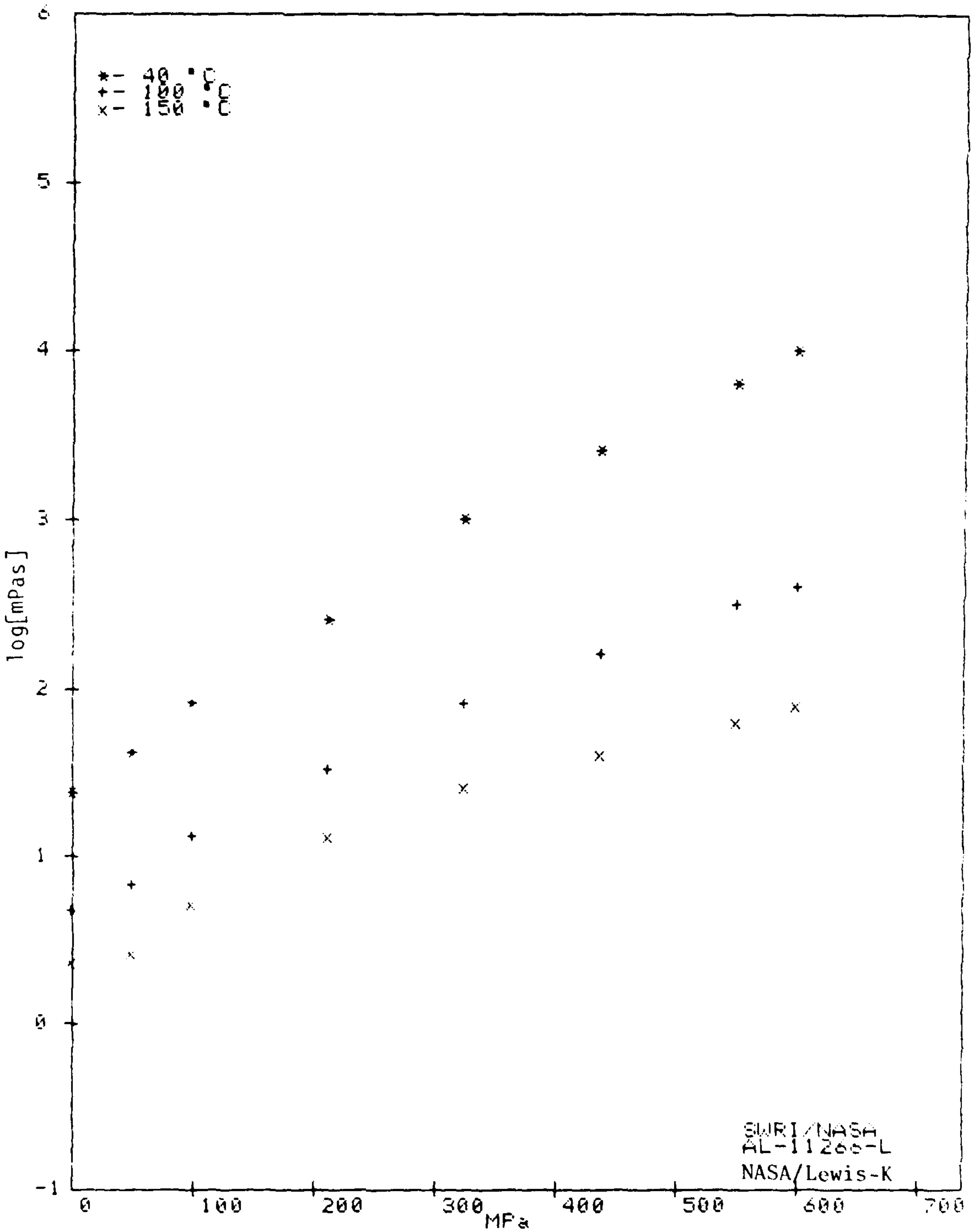
NASA/Lewis - K

temperature	pressure		viscosity
°C	Kpsi	MPa	mPas
40	(1 atm)	0.10	27.70
	6.76	46.62	51.20
	14.85	102.40	95.50
	31.03	213.94	338.00
	47.21	325.49	1170.00
	63.39	437.03	3010.00
	79.57	548.58	7250.00
	87.66	604.35	12500.00
100	(1 atm)	0.10	5.34
	6.76	46.62	8.81
	14.85	102.40	15.20
	31.03	213.94	41.20
	47.21	325.49	102.00
	63.39	437.03	210.00
	79.57	548.58	394.00
	87.66	604.35	540.00
150	(1 atm)	0.10	2.41
	6.76	46.62	3.49
	14.85	102.40	6.19
	31.03	213.94	14.30
	47.21	325.49	29.20
	63.39	437.03	53.20
	79.57	548.58	87.50
	87.66	604.35	106.00

Pressure-Viscosity Coefficients

GPa^-1

Temp °C	α-0T	α-*
40.0	12.25	10.47
100.0	10.15	8.65
150.0	8.96	7.41



*- 40 °C
+- 100 °C
x- 150 °C
log[mPas]
6
5
4
3
2
1
0
-1
0
100
200
300
400
500
600
700
MPa
SWRI/NASA
AL-11266-L
NASA/Lewis-K

APPENDIX D

BOILING POINT DISTRIBUTION DATA

TABLE D-1. NEW TRANSMISSION OILS, BOILING POINT DISTRIBUTION BY GC

AFLRL No.	AL-11252-L	AL-11268-L	AL-11250-L	AL-11254-L	AL-11256-L	AL-11258-L	AL-11260-L	AL-11262-L	AL-11264-L	AL-11270-L	AL-11266-L
Description	A	B	C	D	E	F	G	H	I	J	K
Wt% Off	Temperature, °C										
IBP, 0.5	304	282	372	376	271	324	316	337	360	362	385
1	320	299	376	407	271	350	332	386	362	397	414
5	351	336	422	426	375	412	381	390	424	430	437
10	363	351	433	437	416	417	407	391	442	443	446
15	371	361	440	445	453	421	416	392	446	447	453
20	379	369	447	451	459	423	421	393	452	454	459
25	385	375	454	457	462	426	426	412	455	460	463
30	391	381	457	462	464	429	434	436	461	466	468
35	397	387	463	468	466	432	437	438	468	469	474
40	402	393	470	473	468	448	438	439	470	475	477
45	407	398	477	478	469	472	443	440	476	481	483
50	412	405	489	483	470	477	461	441	483	487	489
55	416	409	513	491	472	481	470	442	490	494	497
60	421	416	548	502	473	483	477	496	513	514	509
65	426	422	(62) 568	538	475	485	481		534	547	547
70	431	428		(69) 568	476	487	485		578	(68) 568	(68) 578
75	437	435			479	489	490				
80	444	444			518	492	501				
85	452	454				498	527				
90	463	466				515	(87) 568				
95	488	488				532					
FBP, 99.5	(97) 518	(98) 518				(97) 568					
% Residue	3.9	2.0	38.9	31.9	20.7	3.1	13.2	40.0	30.1	32.9	32.6

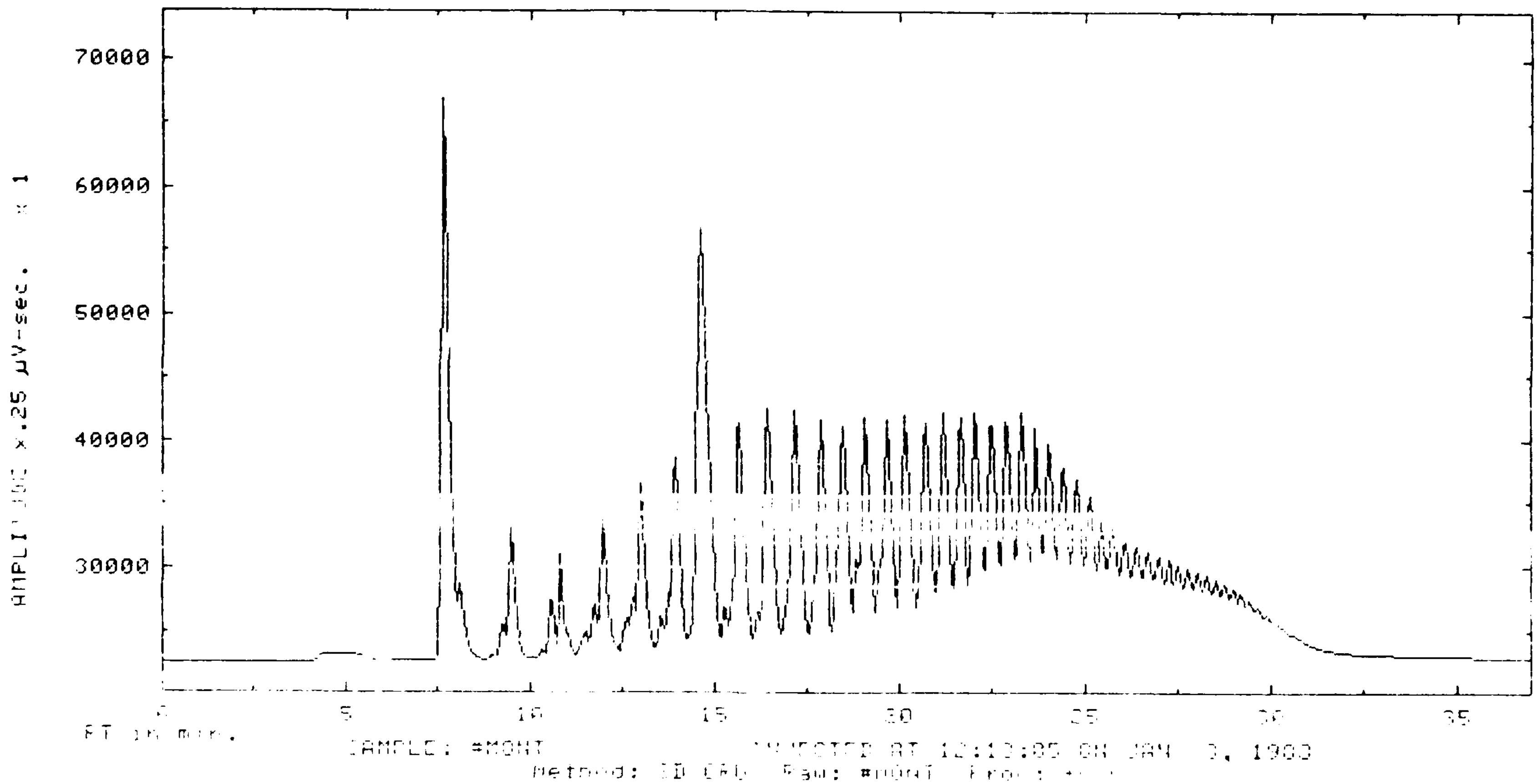


FIGURE D-1. BOILING POINT STANDARD

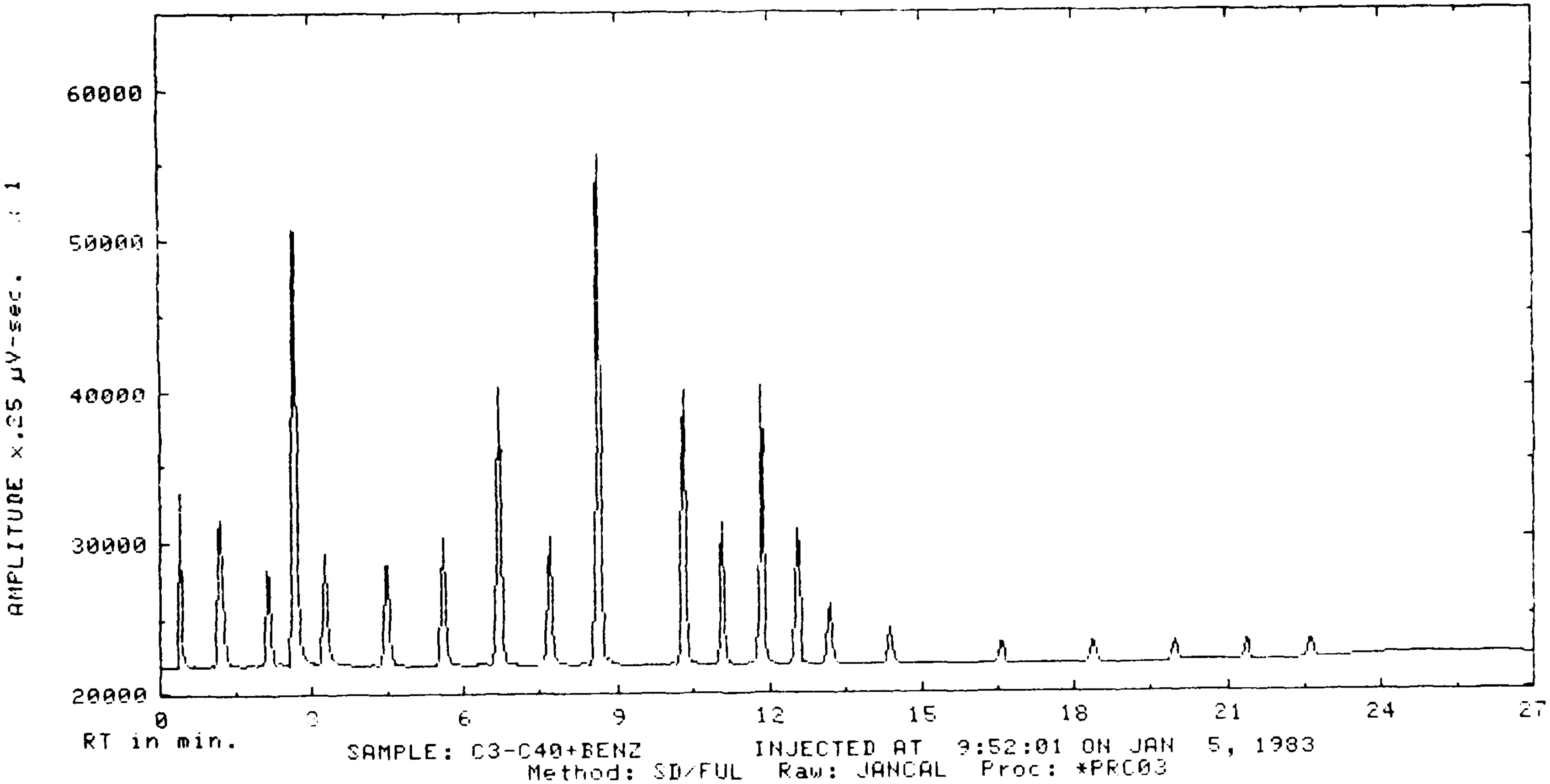


FIGURE D-2. C4-C40 AND BENZENE BOILING POINT STANDARD

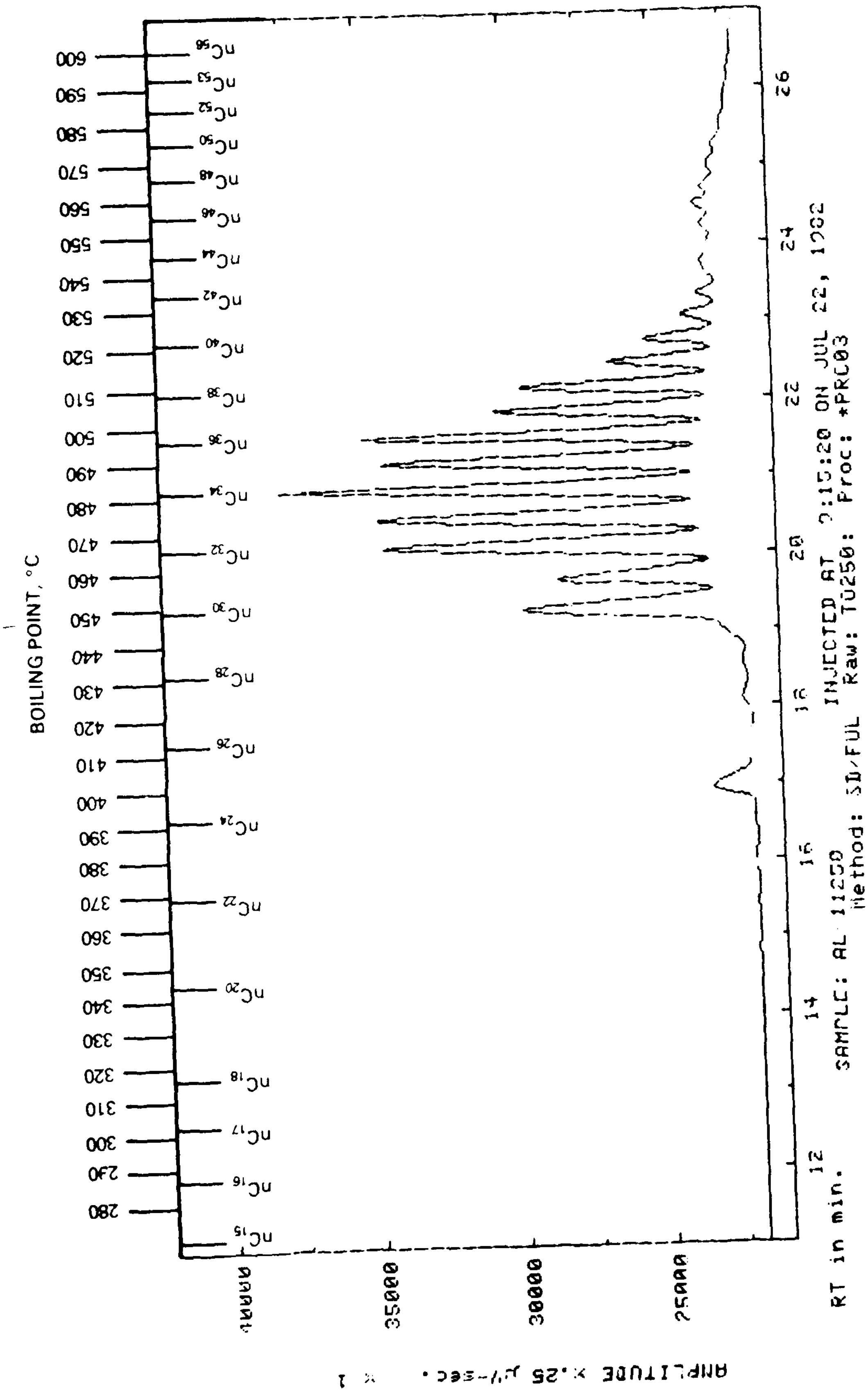

BOILING POINT, °C
280
300
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450
460
470
480
490
500
510
520
530
540
550
560
570
580
590
600
nC_{15}
nC_{16}
nC_{17}
nC_{18}
nC_{20}
nC_{22}
nC_{24}
nC_{26}
nC_{28}
nC_{30}
nC_{32}
nC_{34}
nC_{36}
nC_{38}
nC_{40}
nC_{42}
nC_{44}
nC_{46}
nC_{48}
nC_{50}
nC_{52}
nC_{53}
nC_{56}
35000
30000
25000
AMPLITUDE x.25 µV-sec.
RT in min.
12
14
16
20
22
24
26
SAMPLE: AL 11250
Method: SD/FUL
Raw: TU250:
Proc: *PRC03
ON JUL 22,

FIGURE D-3.

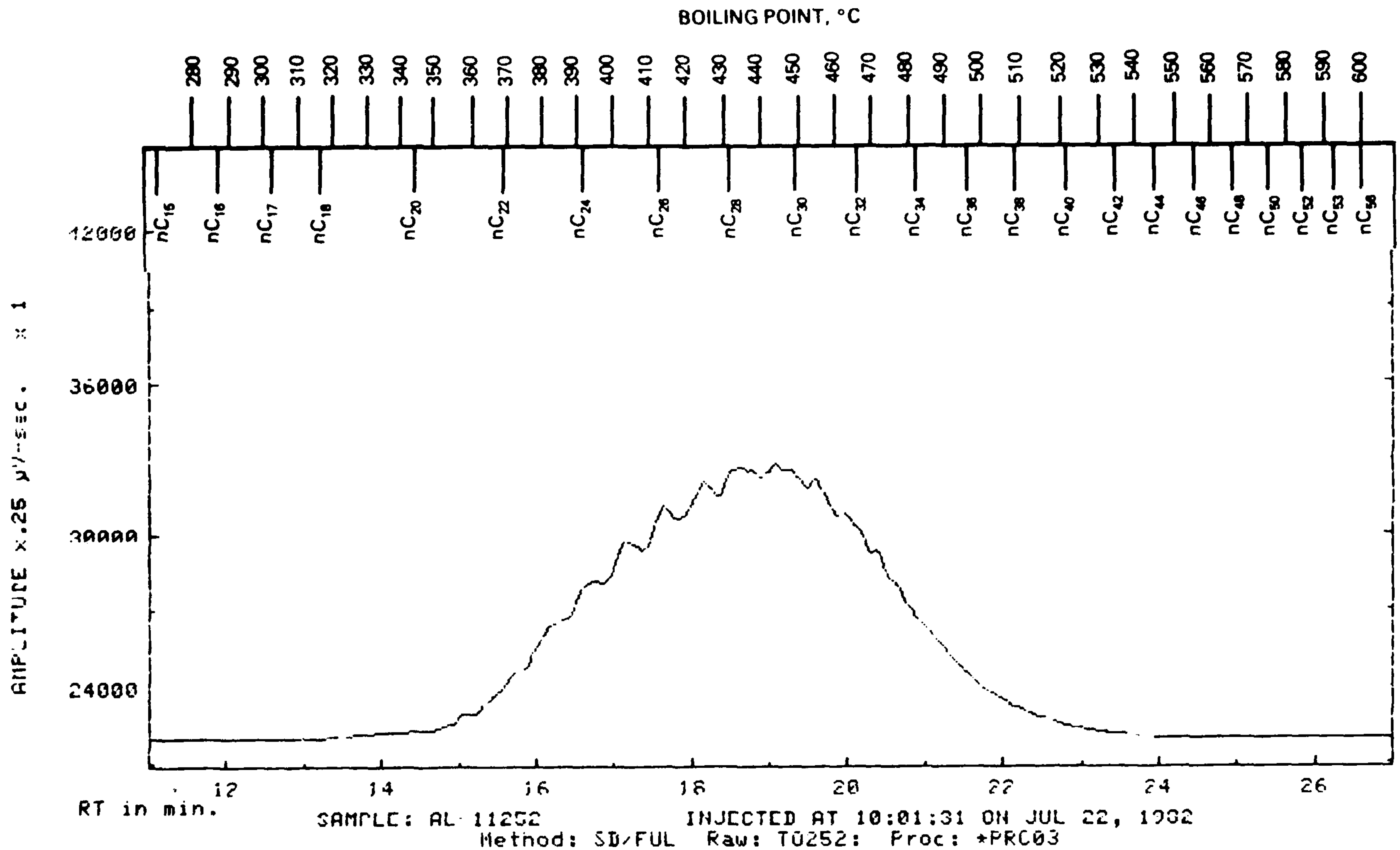
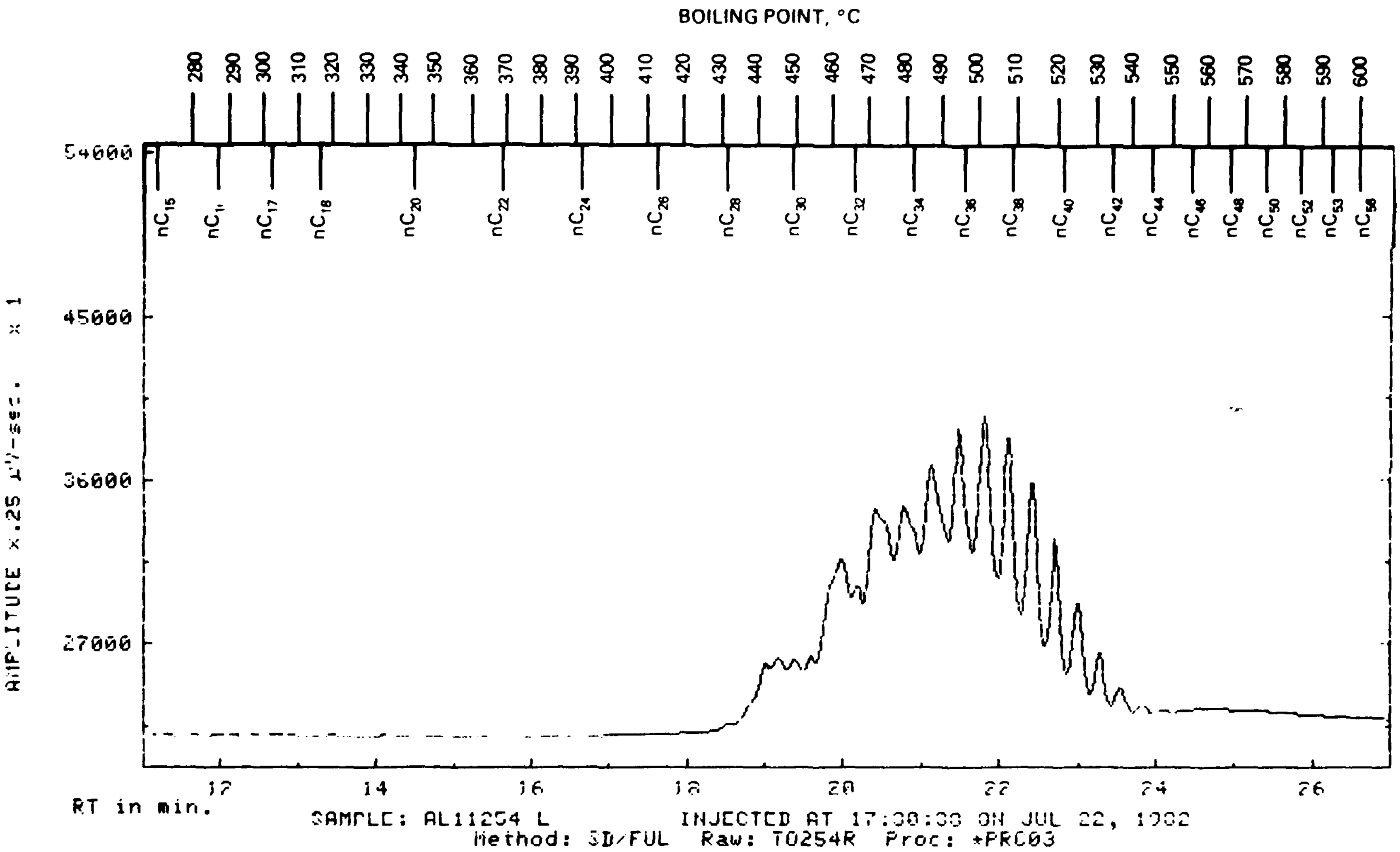
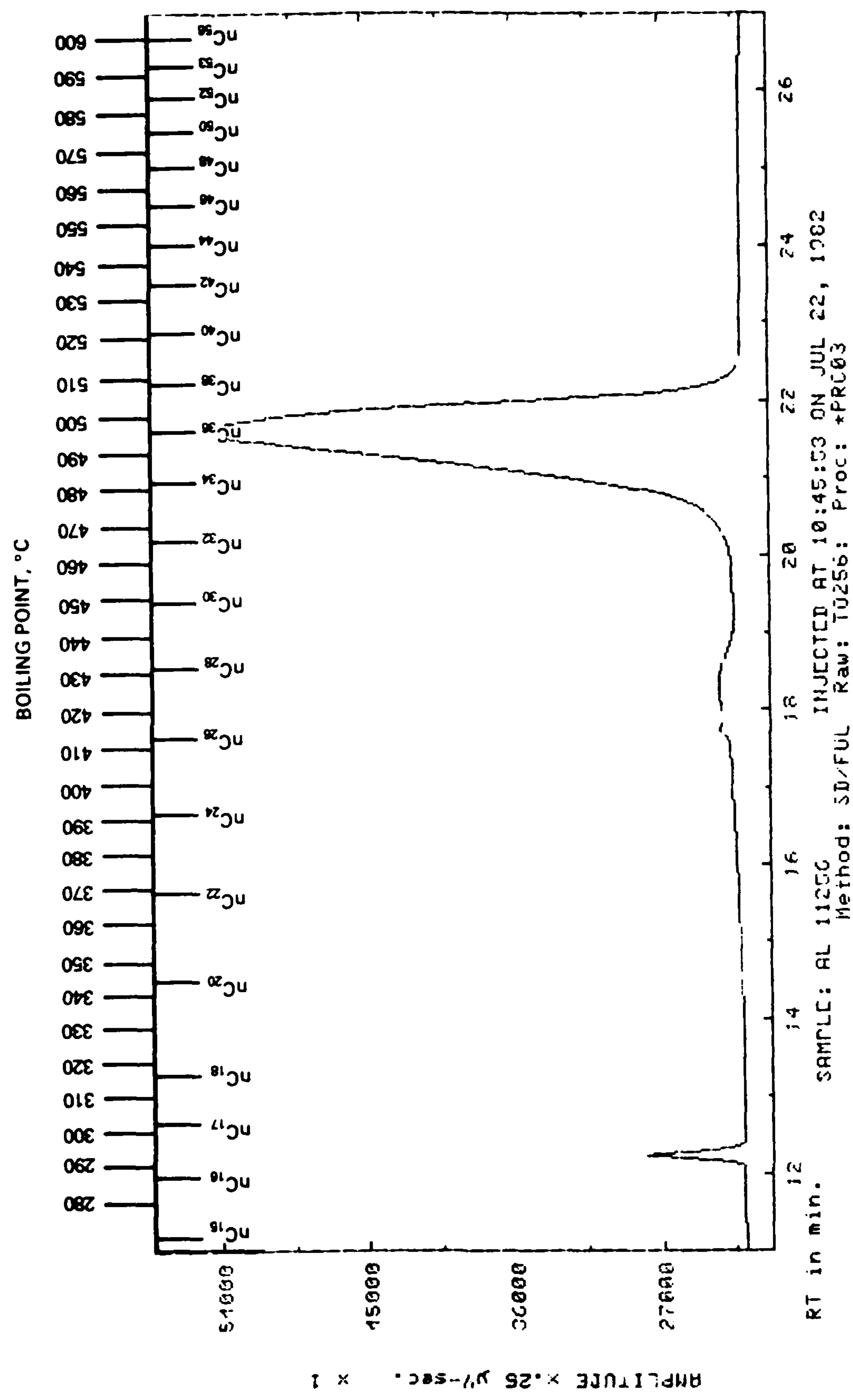

BOILING POINT, °C
280
290
300
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450
460
470
480
490
500
510
520
530
540
550
560
570
580
590
600
nC_{15}
nC_{16}
nC_{17}
nC_{18}
nC_{20}
nC_{22}
nC_{24}
nC_{26}
nC_{28}
nC_{30}
nC_{32}
nC_{34}
nC_{36}
nC_{38}
nC_{40}
nC_{42}
nC_{44}
nC_{46}
nC_{48}
nC_{50}
nC_{52}
nC_{53}
nC_{56}
42000
36000
30000
24000
AMPLITUDE x.25 μV-sec. x 1
12
14
16
18
20
22
24
26
RT in min.
SAMPLE: AL 11252
INJECTED AT 10:01:31 ON JUL 22, 1982
Method: SD/FUL Raw: T0252: Proc: *PRC03

FIGURE D-4.



BOILING POINT, °C
280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500 510 520 530 540 550 560 570 580 590 600
nC15 nC17 nC18 nC20 nC22 nC24 nC26 nC28 nC30 nC32 nC34 nC36 nC38 nC40 nC42 nC44 nC46 nC48 nC50 nC52 nC53 nC56
54000
45000
36000
27000
AMPLITUDE x.25 µV-sec. x 1
12 14 16 18 20 22 24 26
RT in min.
SAMPLE: AL11254 L INJECTED AT 17:30:30 ON JUL 22, 1982
Method: SD/FUL Raw: T0254R Proc: *PRC03

FIGURE D-5.



BOILING POINT, °C
RT in min.
AMPLITUDE x.25 µV-sec. x 1
SAMPLE: AL 11256 INJECTED AT 10:45:53 ON JUL 22, 1982
Method: SD/FUL Raw: TU256: Proc: *PRC03

FIGURE D-6.

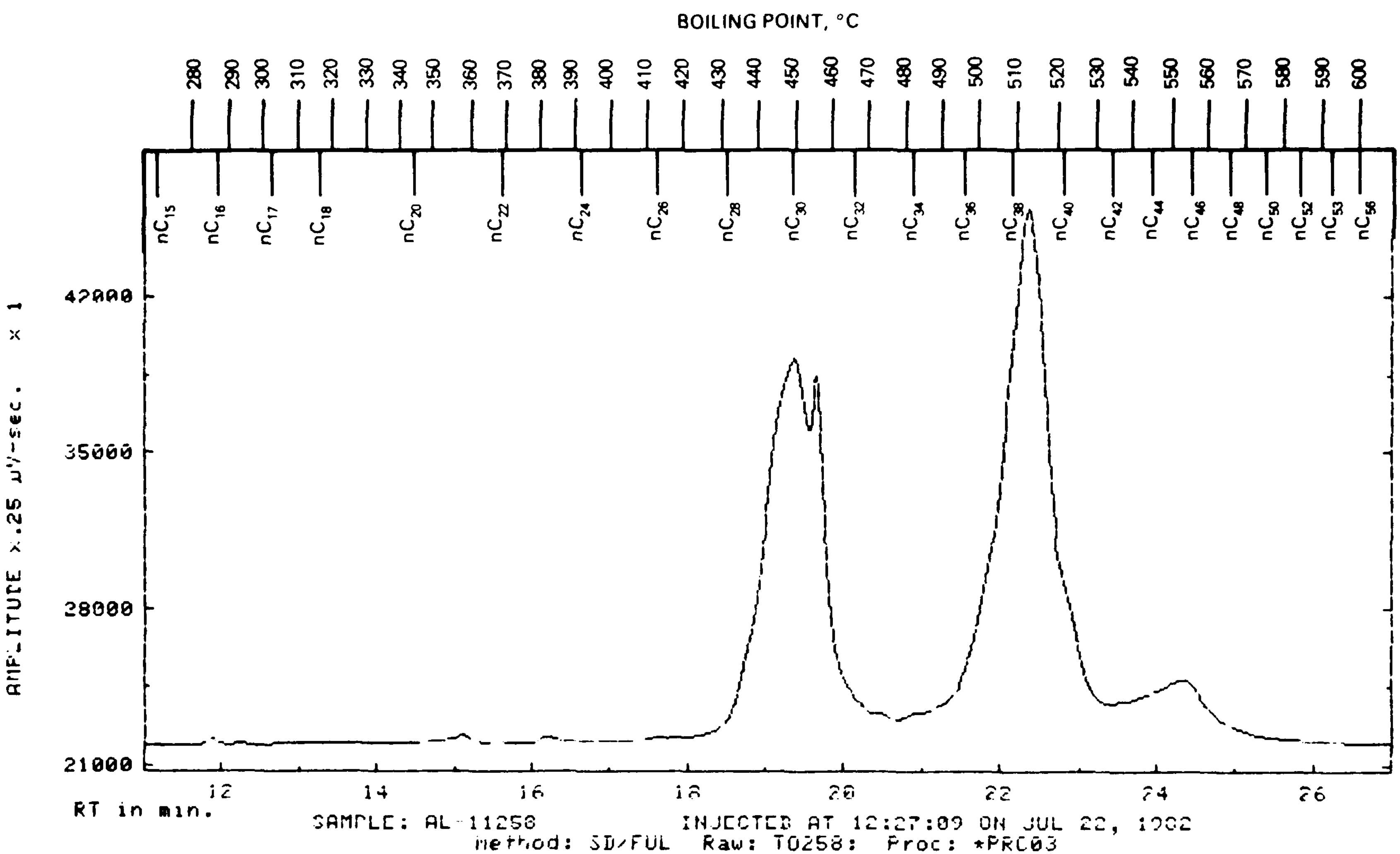

BOILING POINT, °C
280
290
300
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450
460
470
480
490
500
510
520
530
540
550
560
570
580
590
600
nC_{15}
nC_{16}
nC_{17}
nC_{18}
nC_{20}
nC_{22}
nC_{24}
nC_{26}
nC_{28}
nC_{30}
nC_{32}
nC_{34}
nC_{36}
nC_{38}
nC_{40}
nC_{42}
nC_{44}
nC_{46}
nC_{48}
nC_{50}
nC_{52}
nC_{53}
nC_{56}
AMPLITUDE x.25 µV-sec. x 1
42000
35000
28000
21000
12
14
16
18
20
22
24
26
RT in min.
SAMPLE: AL-11258 INJECTED AT 12:27:09 ON JUL 22, 1982
Method: SD/FUL Raw: T0258: Proc: *PRC03

FIGURE D-7.

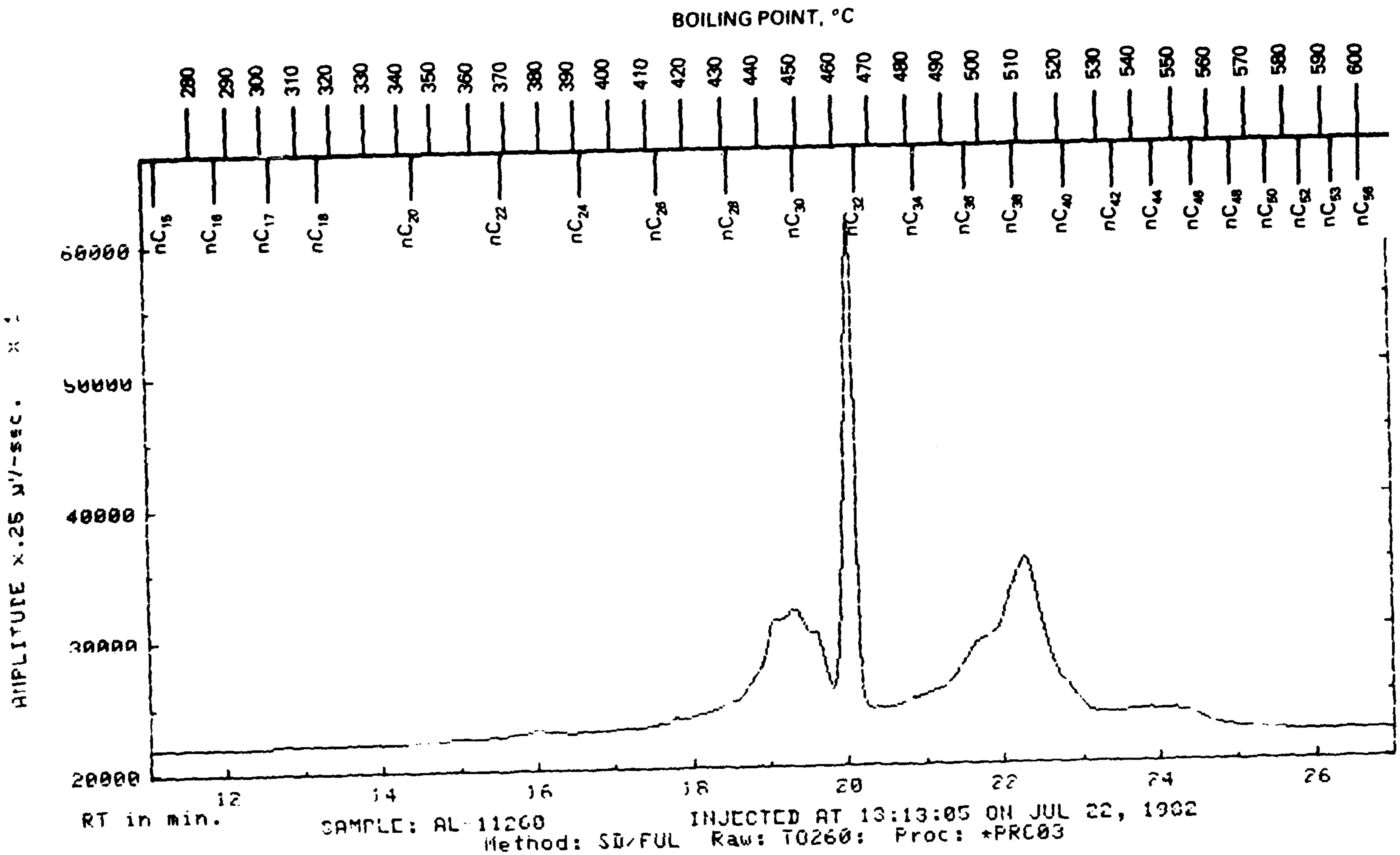

BOILING POINT, °C
280
290
300
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450
460
470
480
490
500
510
520
530
540
550
560
570
580
590
600
nC15
nC16
nC17
nC18
nC20
nC22
nC24
nC26
nC28
nC30
nC32
nC34
nC36
nC38
nC40
nC42
nC44
nC46
nC48
nC50
nC52
nC53
nC56
60000
50000
40000
30000
20000
AMPLITUDE x.25 µV-sec.
12
14
16
18
20
22
24
26
RT in min.
SAMPLE: AL-11260
INJECTED AT 13:13:05 ON JUL 22, 1982
Method: SD/FUL Raw: T0260: Proc: *PRC03

FIGURE D-8.

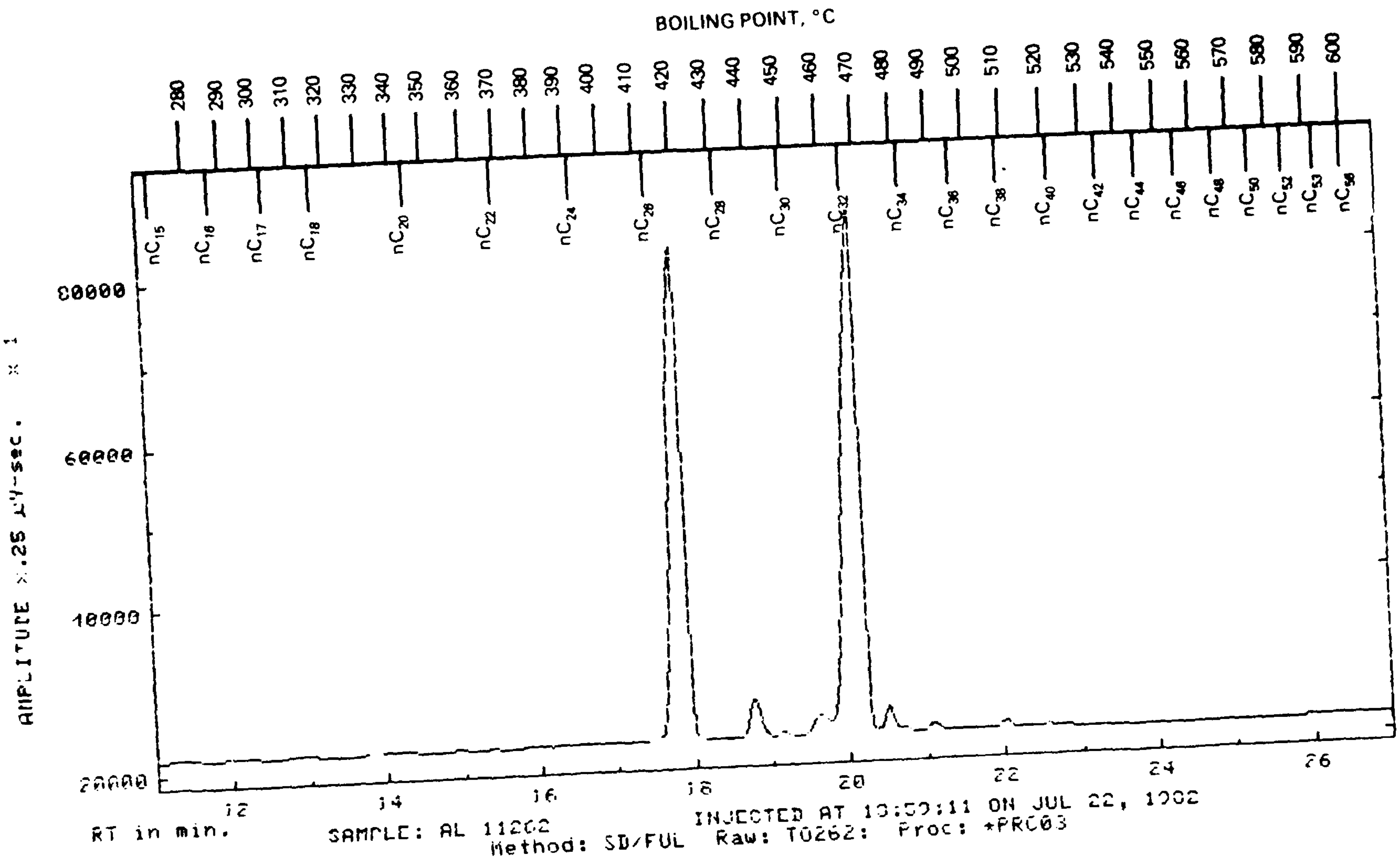

BOILING POINT, °C
280
290
300
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450
460
470
480
490
500
510
520
530
540
550
560
570
580
590
600
nC15
nC16
nC17
nC18
nC20
nC22
nC24
nC26
nC28
nC30
nC32
nC34
nC36
nC38
nC40
nC42
nC44
nC46
nC48
nC50
nC52
nC53
nC56
AMPLITUDE X.25 μV-sec. X 1
80000
60000
40000
20000
12
14
16
18
20
22
24
26
RT in min.
SAMPLE: AL 11262
INJECTED AT 13:53:11 ON JUL 22, 1982
Method: SD/FUL Raw: T0262: Proc: *PRC03

FIGURE D-9.

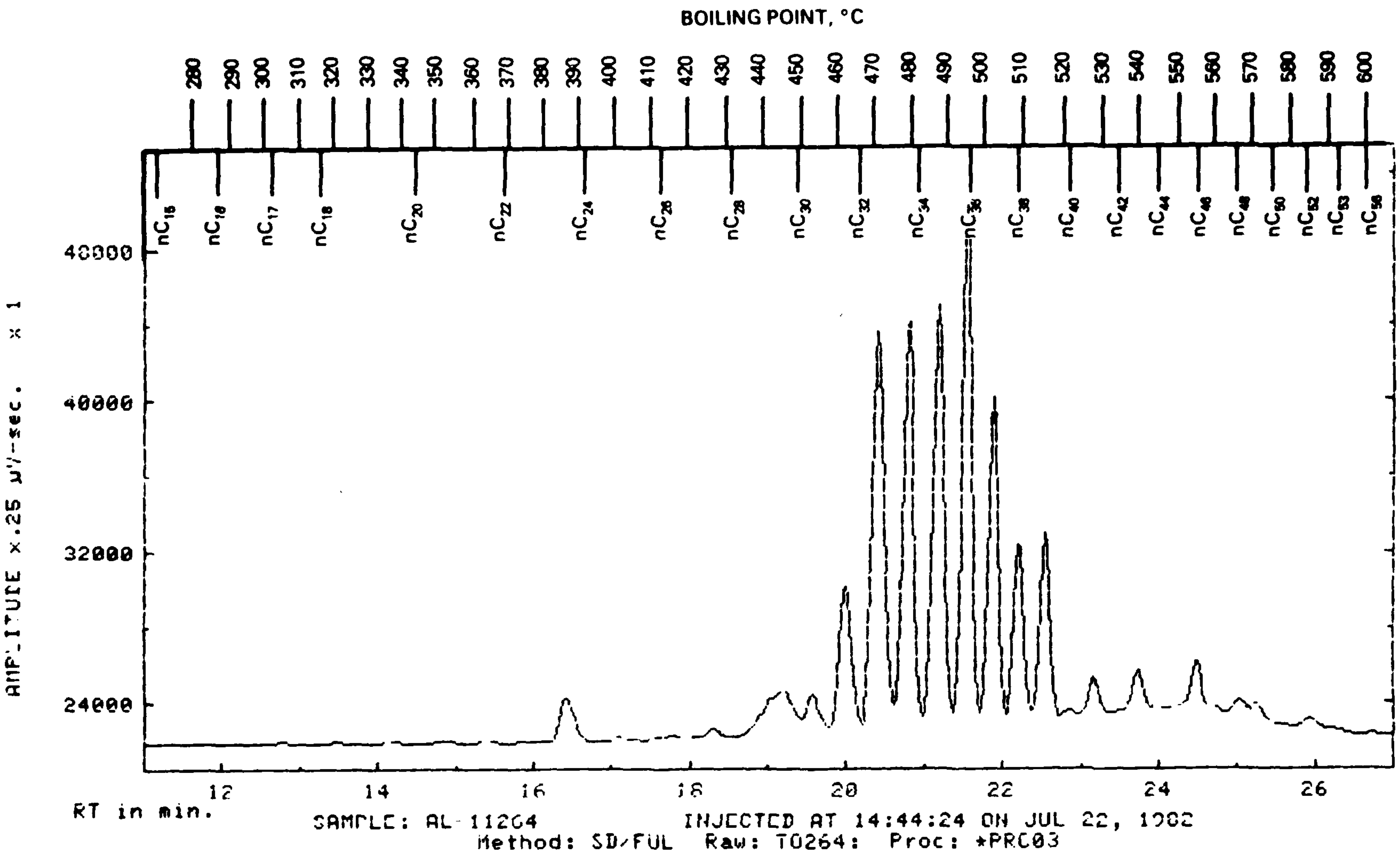

BOILING POINT, °C
280
290
300
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450
460
470
480
490
500
510
520
530
540
550
560
570
580
590
600
nC15
nC16
nC17
nC18
nC20
nC22
nC24
nC26
nC28
nC30
nC32
nC34
nC36
nC38
nC40
nC42
nC44
nC46
nC48
nC50
nC52
nC53
nC56
AMPLITUDE x.25 µV-sec. x 1
48000
40000
32000
24000
12
14
16
18
20
22
24
26
RT in min.
SAMPLE: AL-11264
INJECTED AT 14:44:24 ON JUL 22, 1982
Method: SD/FUL Raw: T0264: Proc: *PRC03

FIGURE D-10.

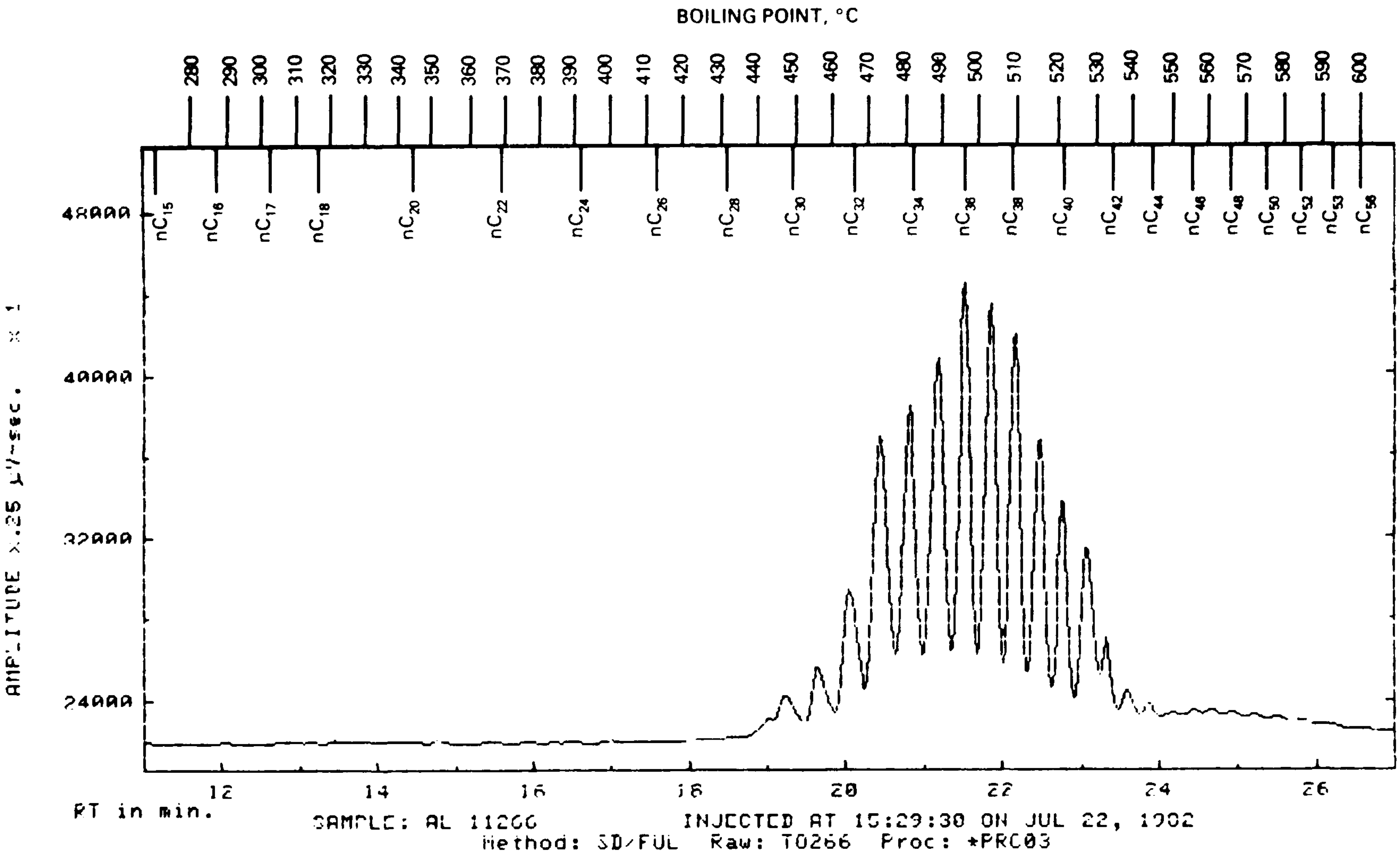

BOILING POINT, °C
280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500 510 520 530 540 550 560 570 580 590 600
nC15 nC16 nC17 nC18 nC20 nC22 nC24 nC26 nC28 nC30 nC32 nC34 nC36 nC38 nC40 nC42 nC44 nC46 nC48 nC50 nC52 nC53 nC56
48000
40000
32000
24000
AMPLITUDE X.25 µV-sec. X 1
12 14 16 18 20 22 24 26
RT in min.
SAMPLE: AL 11266 INJECTED AT 15:29:30 ON JUL 22, 1902
Method: SD/FUL Raw: T0266 Proc: *PRC03

FIGURE D-11.

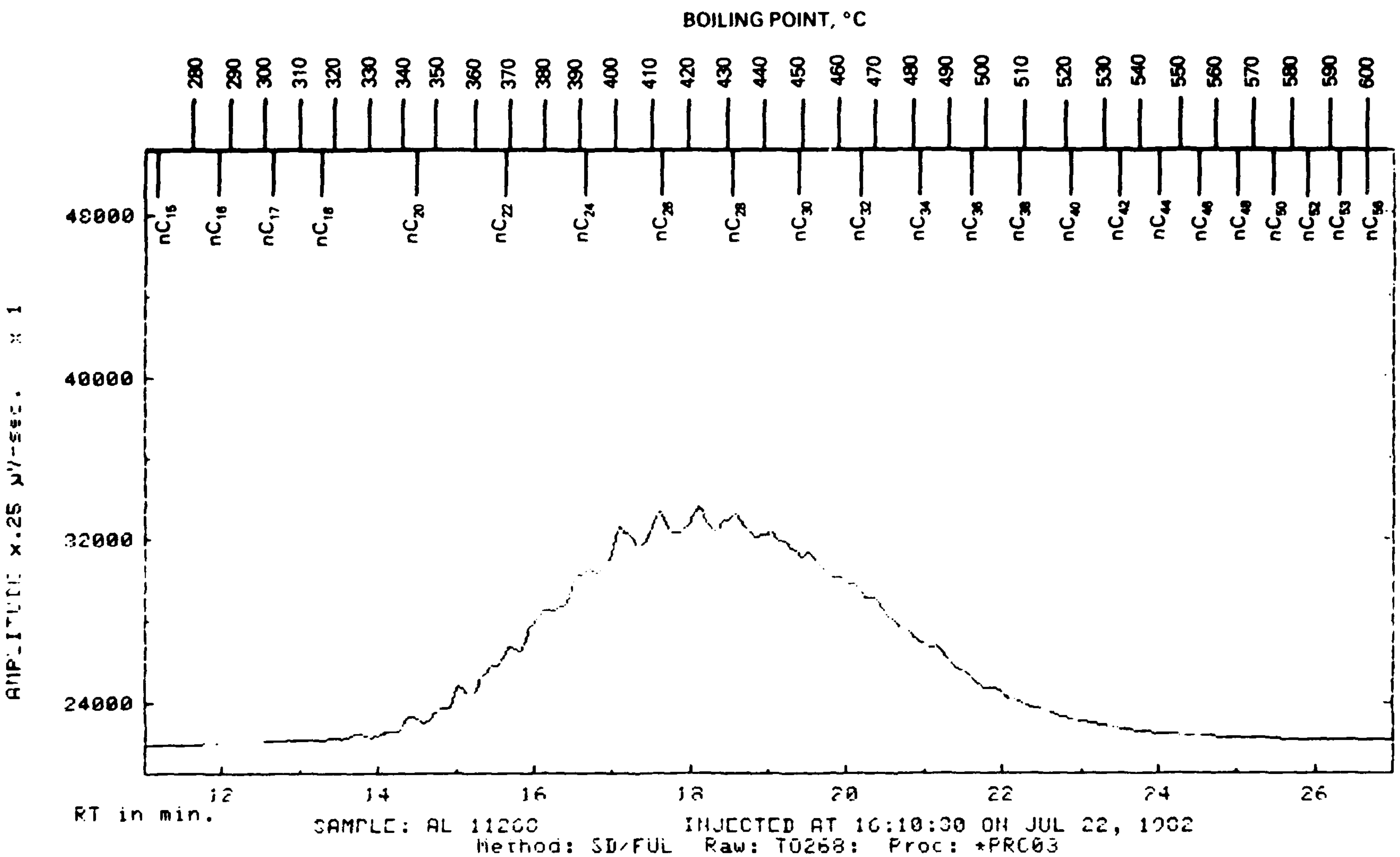

BOILING POINT, °C
280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500 510 520 530 540 550 560 570 580 590 600
nC_{15} nC_{16} nC_{17} nC_{18} nC_{20} nC_{22} nC_{24} nC_{26} nC_{28} nC_{30} nC_{32} nC_{34} nC_{36} nC_{38} nC_{40} nC_{42} nC_{44} nC_{46} nC_{48} nC_{50} nC_{52} nC_{53} nC_{56}
48000
40000
32000
24000
AMPLITUDE x.25 µV-sec. x 1
12 14 16 18 20 22 24 26
RT in min.
SAMPLE: AL 11260 INJECTED AT 16:10:30 ON JUL 22, 1902
Method: SD/FUL Raw: T0268: Proc: *PRC03

FIGURE D-12.

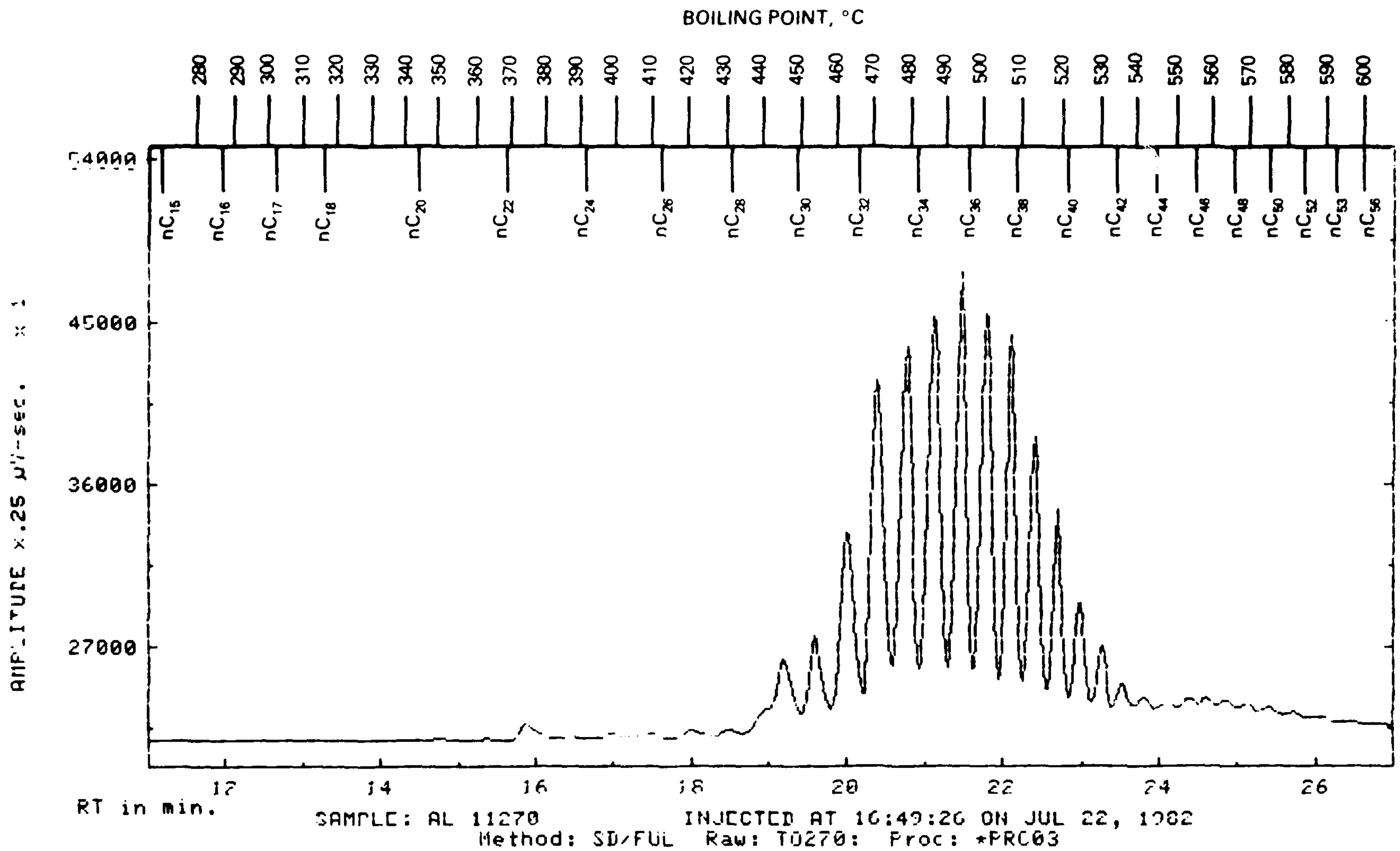

BOILING POINT, °C
280 290 300 310 320 330 340 350 360 370 380 390 400 410 420 430 440 450 460 470 480 490 500 510 520 530 540 550 560 570 580 590 600
nC15 nC16 nC17 nC18 nC20 nC22 nC24 nC26 nC28 nC30 nC32 nC34 nC36 nC38 nC40 nC42 nC44 nC46 nC48 nC50 nC52 nC53 nC56
54000
45000
36000
27000
AMPLITUDE x.25 µV-sec. x 1
12 14 16 18 20 22 24 26
RT in min.
SAMPLE: AL 11270
INJECTED AT 16:49:26 ON JUL 22, 1982
Method: SD/FUL Raw: T0270: Proc: *PRC03

FIGURE D-13.

APPENDIX E

BASESTOCK CHARACTERIZATION STANDARDS

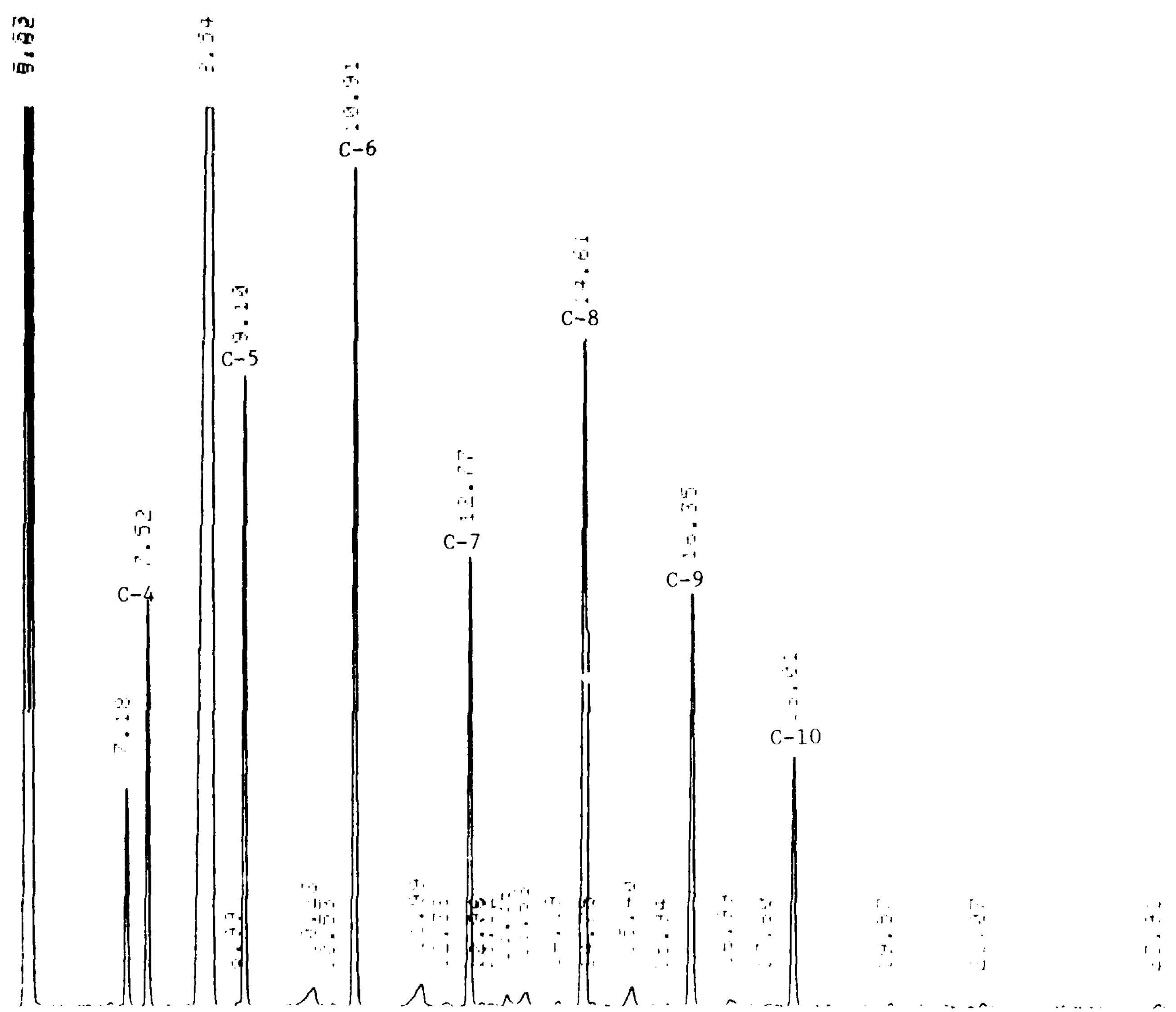


FIGURE F-1. MONO-CARBOXYLIC ACID METHYL ESTER STANDARD

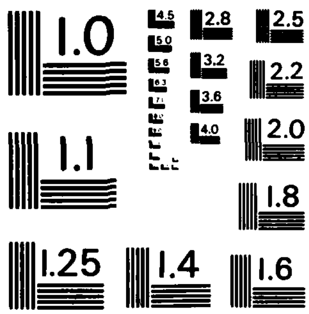
AD A131 945 ADVANCED CHEMICAL CHARACTERIZATION AND PHYSICAL PROPERTIES OF ELEVEN LUBR..(U) SOUTHWEST RESEARCH INST SAN ANTONIO TX ARMY FUELS AND LUBRICA..

UNCLASSIFIED D L PRESENT ET AL. MAR 83 AFLRL-166 F/G 11/8 NL

2/3

END
DATE
FILMED
DTIC

CONT



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

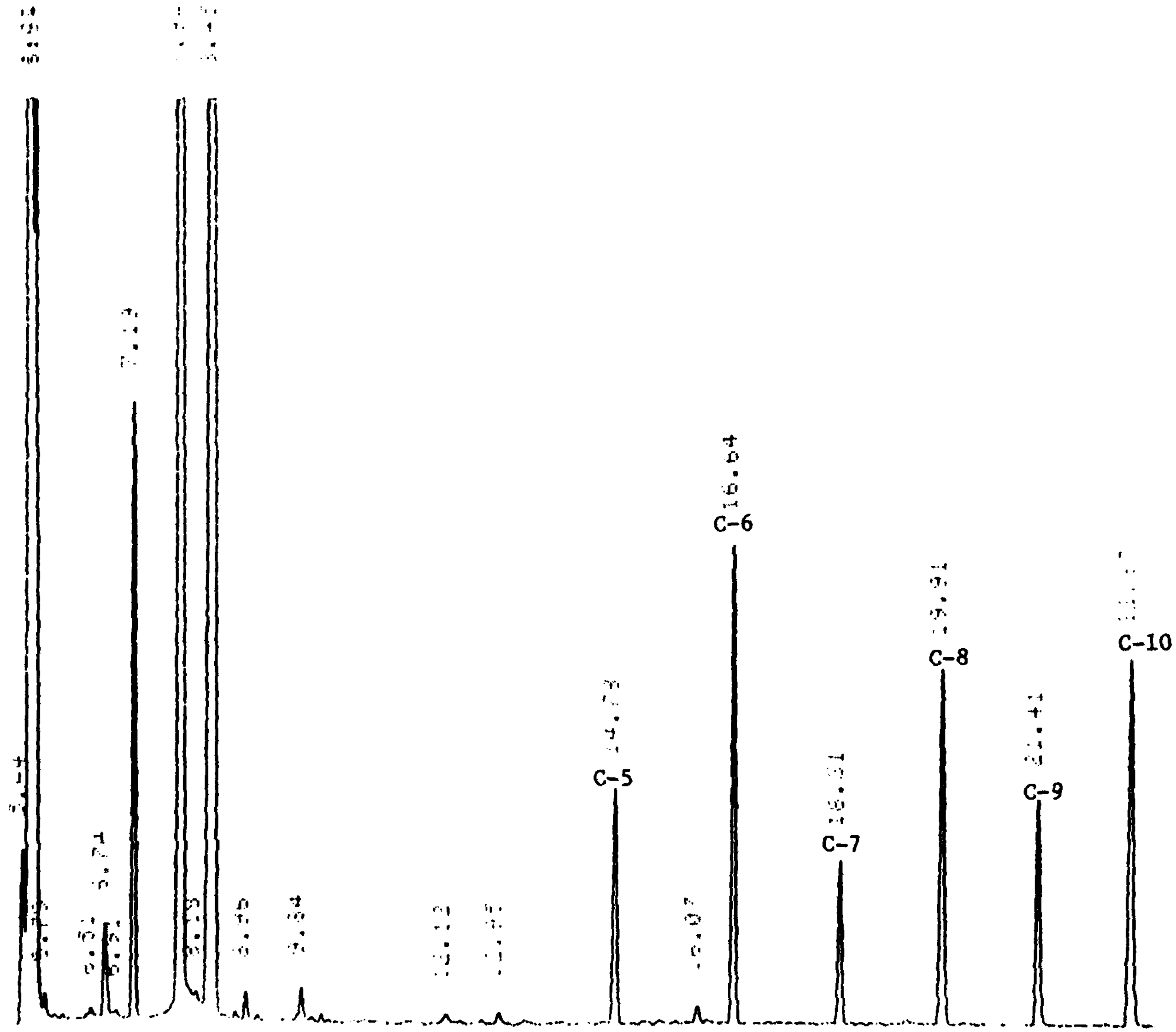


FIGURE E-2. DI CARBOXYLIC ACID METHYL ESTER STANDARDS

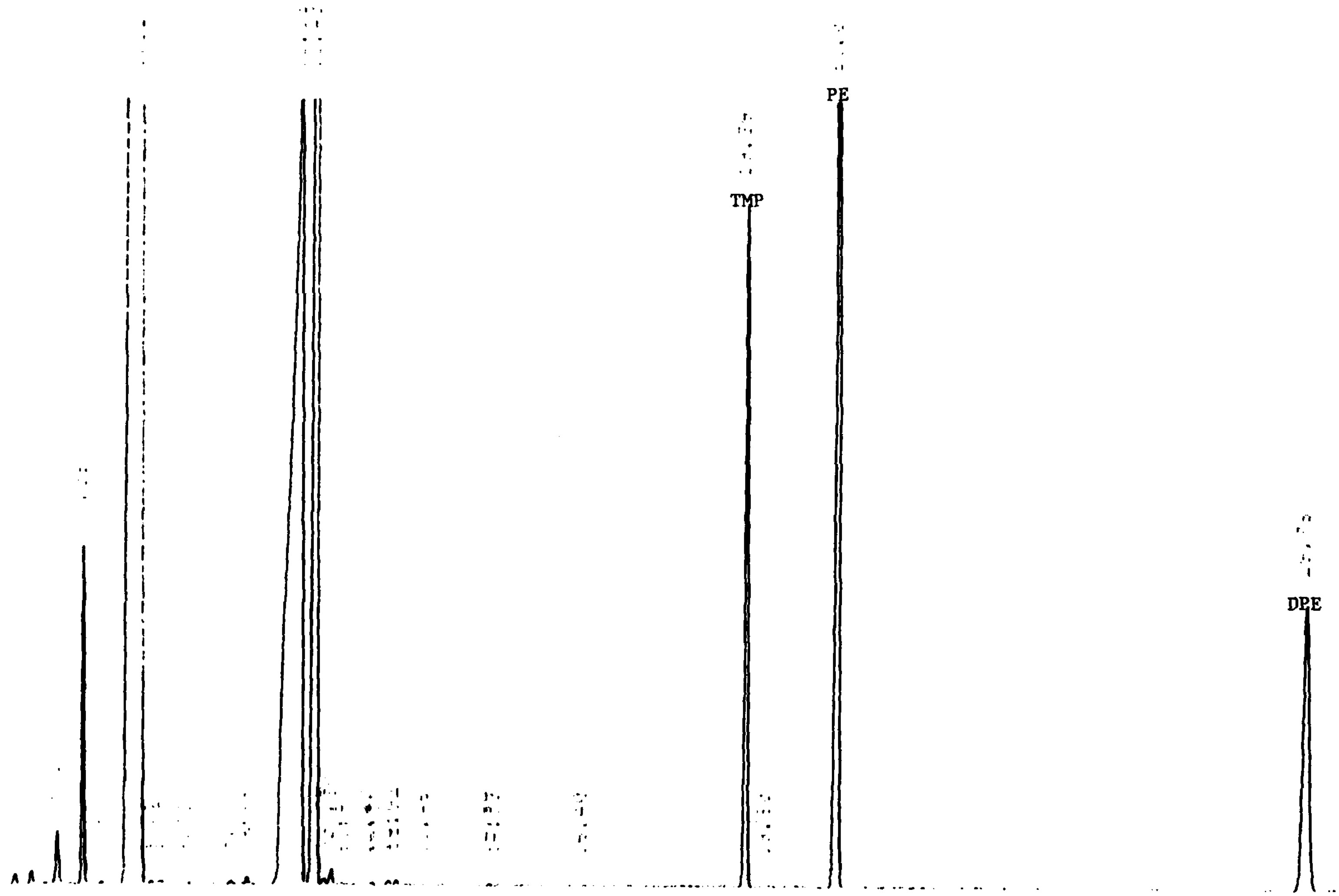


FIGURE E-3. TMP, PE, DPE SILYL DERIVATIVES

FIGURE E-4. PANA DERIVATIVE

FIGURE E-5. P,P'-DIPHENYLDIOCTYLAMINE DERIVATIVE (DPDA)

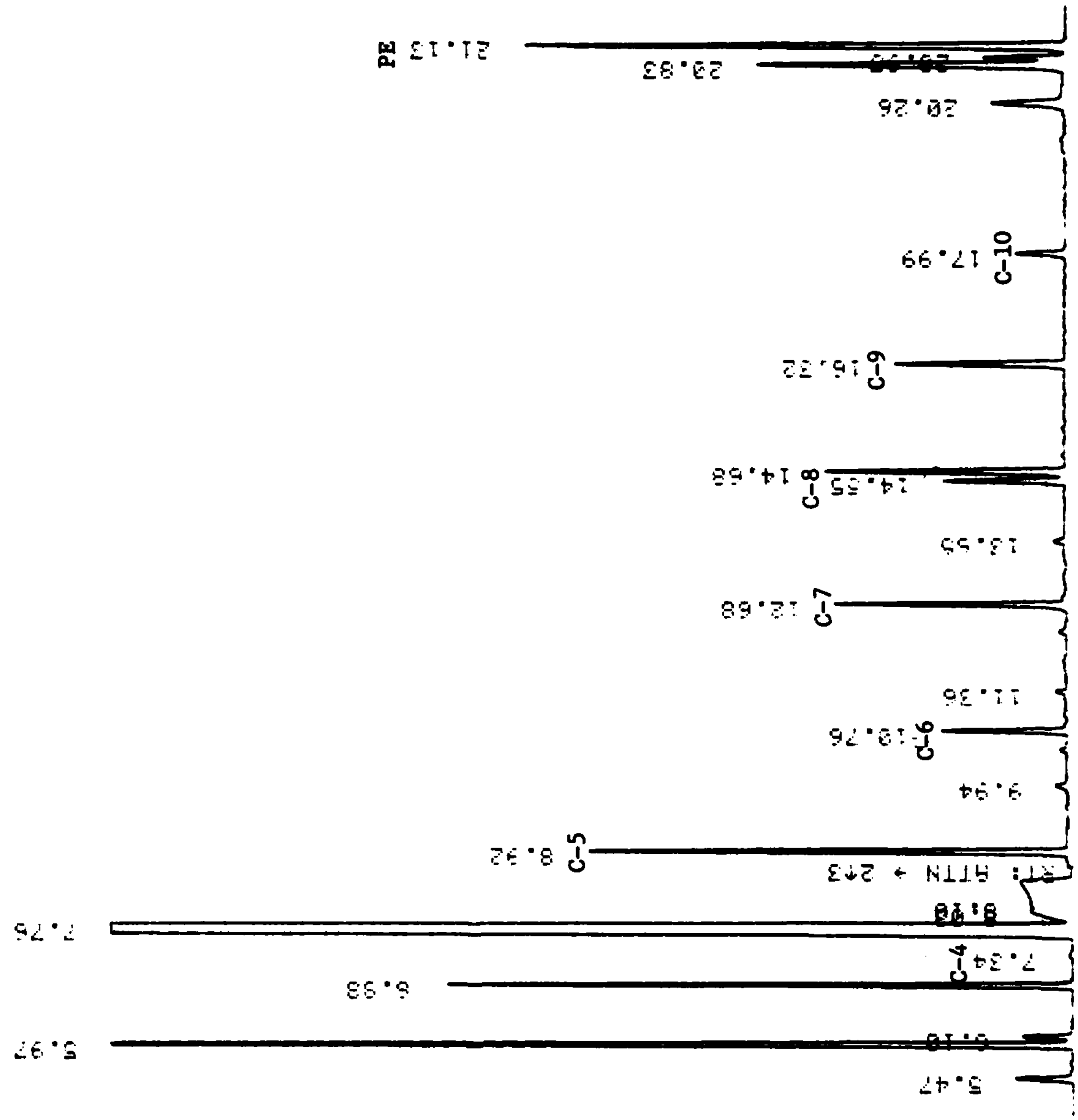


FIGURE E-6. SAMPLE AL-11250-L (NASA C) TRANSESTERIFIED AND SILYLATED

APPENDIX F

BASESTOCK CHARACTERIZATION DATA
WITH DAISY GRAPHS

TABLE F-1. BASESTOCK CHARACTERIZATION SUMMARY

NASA Code	A	B	C	D	E	F	G	H	I	J	K
AFLRL Code	11252	11268	11250	11254	11256	11258	11260	11262	11264	11270	11266
Carboxylic Acids - %	Petroleum-based	Petroleum-based									
C-4			T	9						T	T
C-5			46	18	di-63				13	22	22
C-6			10	13	di-37			T	2	14	16
C-7			17	16			73	50	19	21	24
								35			
C-8			10	24			27	1	30	8	8
C-9			13	16				7	4	23	29
C-10			4	4				5	32	12	1
C-12								2	T		
Alcohols											
TMP							100	100	50		
PE			100	100					50	100	99
DPE											1
MONO-					(C13) 100						
Basestock Type											
Ester			x	x	dibasic		(20%)	x	x	x	x
Petroleum	x	x									
Synthetic Hydrocarbon						x	(80%)				
C30, %						43	38				
C40, %						45	50				
C50, %						12	12				

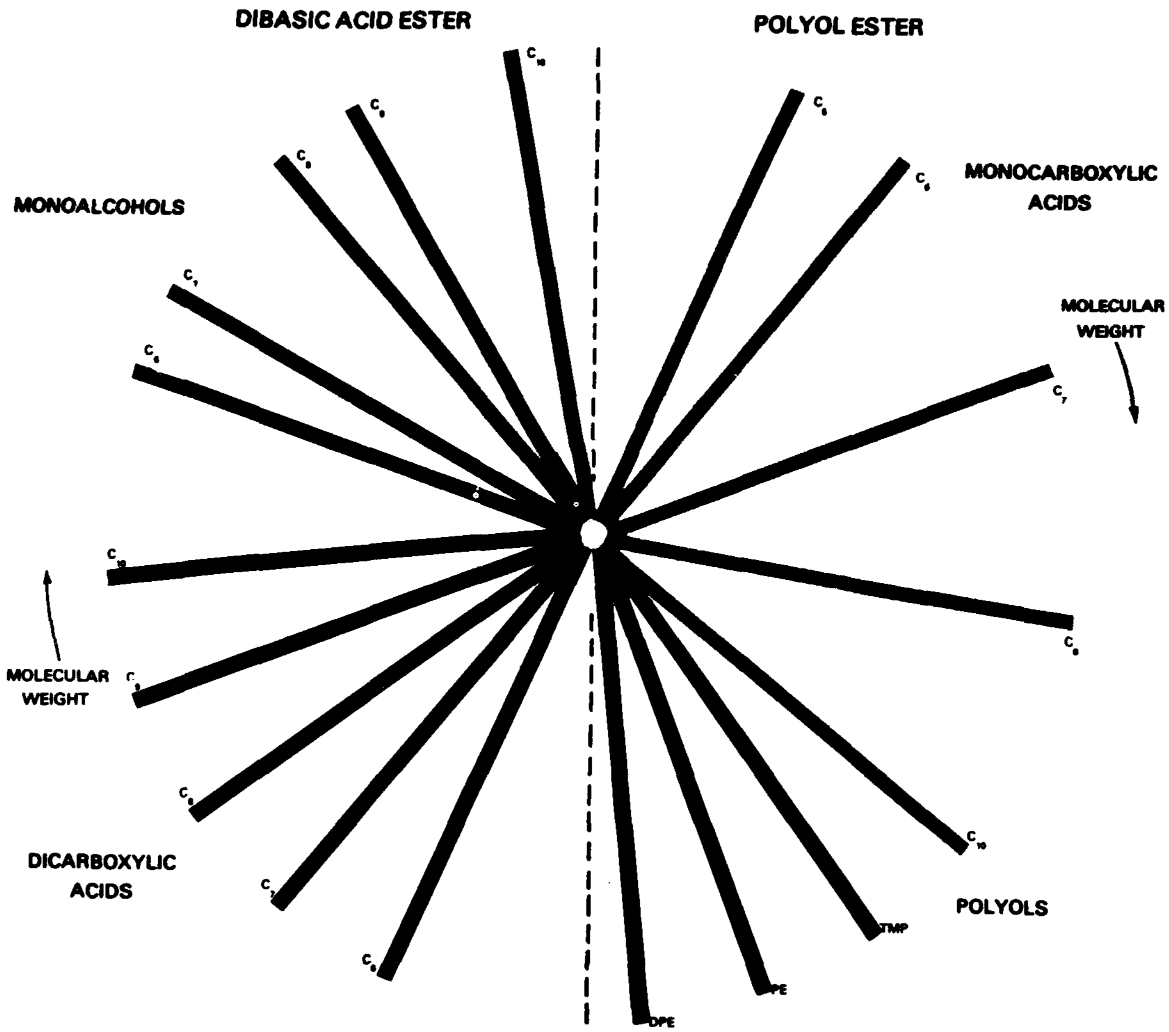


FIGURE F-1. DAISY GRAPH KEY

FIGURE F-2. NASA-C

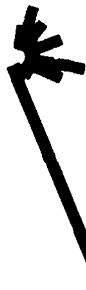


FIGURE F-3. NASA-D

FIGURE F-4. NASA-E

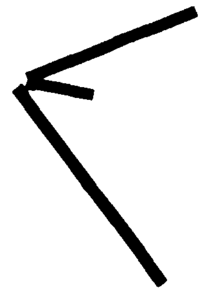


FIGURE F-5. NASA-G

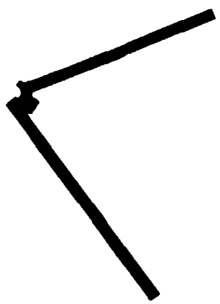


FIGURE F-6. NASA-H

FIGURE F-7. NASA-I

FIGURE F-8. NASA-J

FIGURE F-9. NASA-K

TABLE F-2. BASESTOCK CHARACTERIZATION

SAMPLE DESIGNATION: A
AFLRL No.: AL-11252-L

Chemical Data

• **Polyol Ester Components**

Monocarboxylic Acids		wt%
Butanoic,	C_4	
iso Petanoic,	C_5	
Pentanoic,	C_5	
Hexanoic,	C_6	
iso Heptanoic,	C_7	
Heptanoic,	C_7	
iso Octanoic,	C_8	
Octanoic,	C_8	
Nonanoic,	C_9	
Decanoic,	C_{10}	

• **Dibasic Acid Ester Components**

Dicarboxylic Acids		
Succinic,	C_4	
Glutaric,	C_5	
Adipic,	C_6	
Pimelic,	C_7	
Subaric,	C_8	
Azelaic,	C_9	
Sebacic,	C_{10}	
--,	C_{11}	
--,	C_{12}	

• **Synthetic Hydrocarbon Components**

Hydrocarbon Type		
Triacontane,	C_{30}	
Tetracontane,	C_{40}	
Pentacontane,	C_{50}	

T = Trace

• **Basestock Type**

Basestock Type	wt%
Dibasic Acid Ester	
Polyol Ester	
TMP Ester	
PE Ester	
DPE Ester	
Petroleum	100
Synthetic Hydrocarbon	

Polyols	wt%
Trimethylolpropane, (TMP)	
Pentaerythritol, (PE)	
Dipentaerythritol, (DPE)	

Mono Alcohols		
n-Heptanol	(C_7)	
2-Ethylhexanol	(C_8)	
Octanol	(C_8)	
Nonanol	(C_9)	
Decanol	(C_{10})	
Undecanol	(C_{11})	
Dodecanol	(C_{12})	
Tridecanol	(C_{13})	

TABLE F-3. BASESTOCK CHARACTERIZATION

SAMPLE DESIGNATION: B
AFLRL No.: AL-11268-L

Chemical Data

- **Polyol Ester Components**

Monocarboxylic Acids		wt%
Butanoic,	C_4	
iso Petanoic,	C_5	
Pentanoic,	C_5	
Hexanoic,	C_6	
iso Heptanoic,	C_7	
Heptanoic,	C_7	
iso Octanoic,	C_8	
Octanoic,	C_8	
Nonanoic,	C_9	
Decanoic,	C_{10}	

- **Dibasic Acid Ester Components**

Dicarboxylic Acids		
Succinic,	C_4	
Glutaric,	C_5	
Adipic,	C_6	
Pimelic,	C_7	
Subaric,	C_8	
Azelaic,	C_9	
Sebacic,	C_{10}	
--,	C_{11}	
--,	C_{12}	

- **Synthetic Hydrocarbon Components**

Hydrocarbon Type		
Triacontane,	C_{30}	
Tetracontane,	C_{40}	
Pentacontane,	C_{50}	

- **Basestock Type**

Basestock Type	wt%
Dibasic Acid Ester	
Polyol Ester	
TMP Ester	
PE Ester	
DPE Ester	
Petroleum	100
Synthetic Hyd -arbon	

Polyols	wt%
Trimethylolpropane, (TMP)	
Pentaerythritol, (PE)	
Dipentaerythritol, (DPE)	

Mono Alcohols		
n-Heptanol	(C_7)	
2-Ethylhexanol	(C_8)	
Octanol	(C_8)	
Nonanol	(C_9)	
Decanol	(C_{10})	
Undecanol	(C_{11})	
Dodecanol	(C_{12})	
Tridecanol	(C_{13})	

T = Trace

TABLE F-4. BASESTOCK CHARACTERIZATION

SAMPLE DESIGNATION: C
AFLRL No.: AL-11250-L

Chemical Data

• Polyol Ester Components

Monocarboxylic Acids		wt%
Butanoic,	C_4	T
iso Petanoic,	C_5	
Pentanoic,	C_5	46
Hexanoic,	C_6	10
iso Heptanoic,	C_7	
Heptanoic,	C_7	17
iso Octanoic,	C_8	
Octanoic,	C_8	10
Nonanoic,	C_9	13
Decanoic,	C_{10}	4

• Dibasic Acid Ester Components

Dicarboxylic Acids	
Succinic,	C_4
Glutaric,	C_5
Adipic,	C_6
Pimelic,	C_7
Subaric,	C_8
Azelaic,	C_9
Sebacic,	C_{10}
--,	C_{11}
--,	C_{12}

• Synthetic Hydrocarbon Components

Hydrocarbon Type	
Triacontane,	C_{30}
Tetracontane,	C_{40}
Pentacontane,	C_{50}

T = Trace

• Basestock Type

Basestock Type	wt%
Dibasic Acid Ester	
Polyol Ester	100
TMP Ester	
PE Ester	
DPE Ester	
Petroleum	
Synthetic Hydrocarbon	

Polyols	wt%
Trimethylolpropane, (TMP)	
Pentaerythritol, (PE)	100
Dipentaerythritol, (DPE)	

Mono Alcohols	
n-Heptanol	(C_7)
2-Ethylhexanol	(C_8)
Octanol	(C_8)
Nonanol	(C_9)
Decanol	(C_{10})
Undecanol	(C_{11})
Dodecanol	(C_{12})
Tridecanol	(C_{13})

TABLE F-5. BASESTOCK CHARACTERIZATION

SAMPLE DESIGNATION: D
AFLRL No.: AL-11254-L

Chemical Data

- **Polyol Ester Components**

Monocarboxylic Acids		wt%
Butanoic,	C_4	9
iso Petanoic,	C_5	
Pentanoic,	C_5	18
Hexanoic,	C_6	13
iso Heptanoic,	C_7	
Heptanoic,	C_7	16
iso Octanoic,	C_8	
Octanoic,	C_8	24
Nonanoic,	C_9	16
Decanoic,	C_{10}	4

- **Dibasic Acid Ester Components**

Dicarboxylic Acids	
Succinic,	C_4
Glutaric,	C_5
Adipic,	C_6
Pimelic,	C_7
Subaric,	C_8
Azelaic,	C_9
Sebacic,	C_{10}
--,	C_{11}
--,	C_{12}

- **Synthetic Hydrocarbon Components**

Hydrocarbon Type	
Triacontane,	C_{30}
Tetracontane,	C_{40}
Pentacontane,	C_{50}

- **Basestock Type**

Basestock Type	wt%
Dibasic Acid Ester	
Polyol Ester	100
TMP Ester	
PE Ester	
DPE Ester	
Petroleum	
Synthetic Hydrocarbon	

Polyols	wt%
Trimethylolpropane, (TMP)	
Pentaerythritol, (PE)	100
Dipentaerythritol, (DPE)	

Mono Alcohols	
n-Heptanol	(C_7)
2-Ethylhexanol	(C_8)
Octanol	(C_8)
Nonanol	(C_9)
Decanol	(C_{10})
Undecanol	(C_{11})
Dodecanol	(C_{12})
Tridecanol	(C_{13})

T = Trace

TABLE F-6. BASESTOCK CHARACTERIZATION

SAMPLE DESIGN'TION: E
AFLRL No.: AL-11256-L

Chemical Data

- **Polyol Ester Components**

Monocarboxylic Acids		wt%
Butanoic,	C_4	
iso Petanoic,	C_5	
Pentanoic,	C_5	
Hexanoic,	C_6	
iso Heptanoic,	C_7	
Heptanoic,	C_7	
iso Octanoic,	C_8	
Octanoic,	C_8	
Nonanoic,	C_9	
Decanoic,	C_{10}	

- **Dibasic Acid Ester Components**

Dicarboxylic Acids		
Succinic,	C_4	
Glutaric,	C_5	63
Adipic,	C_6	37
Pimelic,	C_7	
Subaric,	C_8	
Azelaic,	C_9	
Sebacic,	C_{10}	
--,	C_{11}	
--,	C_{12}	

- **Synthetic Hydrocarbon Components**

Hydrocarbon Type	
Triacontane,	C_{30}
Tetracontane,	C_{40}
Pentacontane,	C_{50}

- **Basestock Type**

Basestock Type	wt%
Dibasic Acid Ester	100
Polyol Ester	
TMP Ester	
PE Ester	
DPE Ester	
Petroleum	
Synthetic Hydrocarbon	

Polyols	wt%
Trimethylolpropane, (TMP)	
Pentaerythritol, (PE)	
Dipentaerythritol, (DPE)	

Mono Alcohols		
n-Heptanol	(C_7)	
2-Ethylhexanol	(C_8)	
Octanol	(C_8)	
Nonanol	(C_9)	
Decanol	(C_{10})	
Undecanol	(C_{11})	
Dodecanol	(C_{12})	
Tridecanol	(C_{13})	100

T = Trace

TABLE F-7. BASESTOCK CHARACTERIZATION

SAMPLE DESIGNATION: F
AFLRL No.: AL-11258-L

Chemical Data

- **Polyol Ester Components**

Monocarboxylic Acids		wt%
Butanoic,	C_4	
iso Petanoic,	C_5	
Pentanoic,	C_5	
Hexanoic,	C_6	
iso Heptanoic,	C_7	
Heptanoic,	C_7	
iso Octanoic,	C_8	
Octanoic,	C_8	
Nonanoic,	C_9	
Decanoic,	C_{10}	

- **Dibasic Acid Ester Components**

Dicarboxylic Acids		
Succinic,	C_4	
Glutaric,	C_5	
Adipic,	C_6	
Pimelic,	C_7	
Subaric,	C_8	
Azelaic,	C_9	
Sebacic,	C_{10}	
--,	C_{11}	
--,	C_{12}	

- **Synthetic Hydrocarbon Components**

Hydrocarbon Type		
Triacontane,	C_{30}	43
Tetracontane,	C_{40}	45
Pentacontane,	C_{50}	12

- **Basestock Type**

Basestock Type	wt%
Dibasic Acid Ester	
Polyol Ester	
TMP Ester	
PE Ester	
DPE Ester	
Petroleum	
Syn. Hydrocarbon	100

Polyols	wt%
Trimethylolpropane, (TMP)	
Pentaerythritol, (PE)	
Dipentaerythritol, (DPE)	

Mono Alcohols		
n-Heptanol	(C_7)	
2-Ethylhexanol	(C_8)	
Octanol	(C_8)	
Nonanol	(C_9)	
Decanol	(C_{10})	
Undecanol	(C_{11})	
Dodecanol	(C_{12})	
Tridecanol	(C_{13})	

T = Trace

TABLE F-8. BASESTOCK CHARACTERIZATION

SAMPLE DESIGNATION: G
AFLRL No.: AL-11260-L

Chemical Data

- **Polyol Ester Components**

Monocarboxylic Acids		wt%
Butanoic,	C_4	
iso Petanoic,	C_5	
Pentanoic,	C_5	
Hexanoic,	C_6	
iso Heptanoic,	C_7	
Heptanoic,	C_7	73
iso Octanoic,	C_8	
Octanoic,	C_8	27
Nonanoic,	C_9	
Decanoic,	C_{10}	

- **Dibasic Acid Ester Components**

Dicarboxylic Acids		
Succinic,	C_4	
Glutaric,	C_5	
Adipic,	C_6	
Pimelic,	C_7	
Subaric,	C_8	
Azelaic,	C_9	
Sebacic,	C_{10}	
--,	C_{11}	
--,	C_{12}	

- **Synthetic Hydrocarbon Components**

Hydrocarbon Type		
Triacontane,	C_{30}	38
Tetracontane,	C_{40}	50
Pentacontane,	C_{50}	12

- **Basestock Type**

Basestock Type	wt%
Dibasic Acid Ester	
Polyol Ester	20
TMP Ester	
PE Ester	
DPE Ester	
Petroleum	
Syn. Hydrocarbon	80

Polyols	wt%
Trimethylolpropane, (TMP)	100
Pentaerythritol, (PE)	
Dipentaerythritol, (DPE)	

Mono Alcohols	
n-Heptanol	(C_7)
2-Ethylhexanol	(C_8)
Octanol	(C_8)
Nonanol	(C_9)
Decanol	(C_{10})
Undecanol	(C_{11})
Dodecanol	(C_{12})
Tridecanol	(C_{13})

T = Trace

TABLE F-9. BASESTOCK CHARACTERIZATION

SAMPLE DESIGNATION: H
AFLRL No.: AL-11262-L

Chemical Data

• Polyol Ester Components

Monocarboxylic Acids		wt%
Butanoic,	C_4	
iso Petanoic,	C_5	
Pentanoic,	C_5	
Hexanoic,	C_6	T
iso Heptanoic,	C_7	35
Heptanoic,	C_7	50
iso Octanoic,	C_8	
Octanoic,	C_8	1
Nonanoic,	C_9	7
Decanoic,	C_{10}	5
Dodeconoil	C_{12}	2

• Dibasic Acid Ester Components

Dicarboxylic Acids	
Succinic,	C_4
Glutaric,	C_5
Adipic,	C_6
Pimelic,	C_7
Subaric,	C_8
Azelaic,	C_9
Sebacic,	C_{10}
--,	C_{11}
--,	C_{12}

• Synthetic Hydrocarbon Components

Hydrocarbon Type	
Triacontane,	C_{30}
Tetracontane,	C_{40}
Pentacontane,	C_{50}

• Basestock Type

Basestock Type	wt%
Dibasic Acid Ester	
Polyol Ester	100
TMP Ester	
PE Ester	
DPE Ester	
Petroleum	
Synthetic Hydrocarbon	

Polyols	wt%
Trimethylolpropane, (TMP)	100
Pentaerythritol, (PE)	
Dipentaerythritol, (DPE)	

Mono Alcohols	
n-Heptanol	(C_7)
2-Ethylhexanol	(C_8)
Octanol	(C_8)
Nonanol	(C_9)
Decanol	(C_{10})
Undecanol	(C_{11})
Dodecanol	(C_{12})
Tridecanol	(C_{13})

T = Trace

TABLE F-10. BASESTOCK CHARACTERIZATION

SAMPLE DESIGNATION: I
AFLRL No.: AL-11264-L

Chemical Data

• Polyol Ester Components

Monocarboxylic Acids		wt%
Butanoic,	C_4	
iso Petanoic,	C_5	
Pentanoic,	C_5	13
Hexanoic,	C_6	2
iso Heptanoic,	C_7	
Heptanoic,	C_7	19
iso Octanoic,	C_8	
Octanoic,	C_8	30
Nonanoic,	C_9	4
Decanoic,	C_{10}	32
Dodeconoil	C_{12}	T

• Dibasic Acid Ester Components

Dicarboxylic Acids	
Succinic,	C_4
Glutaric,	C_5
Adipic,	C_6
Pimelic,	C_7
Subaric,	C_8
Azelaic,	C_9
Sebacic,	C_{10}
--,	C_{11}
--,	C_{12}

• Synthetic Hydrocarbon Components

Hydrocarbon Type	
Triacontane,	C_{30}
Tetracontane,	C_{40}
Pentacontane,	C_{50}

T = Trace

• Basestock Type

Basestock Type	wt%
Dibasic Acid Ester	
Polyol Ester	100
TMP Ester	50
PE Ester	50
DPE Ester	
Petroleum	
Synthetic Hydrocarbon	

Polyols	wt%
Trimethylolpropane, (TMP)	55
Pentaerythritol, (PE)	45
Dipentaerythritol, (DPE)	

Mono Alcohols	
n-Heptanol	(C_7)
2-Ethylhexanol	(C_8)
Octanol	(C_8)
Nonanol	(C_9)
Decanol	(C_{10})
Undecanol	(C_{11})
Dodecanol	(C_{12})
Tridecanol	(C_{13})

TABLE F-11. BASESTOCK CHARACTERIZATION

SAMPLE DESIGNATION: J
AFLRL No.: AL-11270-L

Chemical Data

- **Polyol Ester Components**

Monocarboxylic Acids		wt%
Butanoic,	C_4	T
iso Petanoic,	C_5	
Pentanoic,	C_5	22
Hexanoic,	C_6	14
iso Heptanoic,	C_7	
Heptanoic,	C_7	21
iso Octanoic,	C_8	
Octanoic,	C_8	8
Nonanoic,	C_9	23
Decanoic,	C_{10}	12

- **Dibasic Acid Ester Components**

Dicarboxylic Acids	
Succinic,	C_4
Glutaric,	C_5
Adipic,	C_6
Pimelic,	C_7
Subaric,	C_8
Azelaic,	C_9
Sebacic,	C_{10}
--,	C_{11}
--,	C_{12}

- **Synthetic Hydrocarbon Components**

Hydrocarbon Type	
Triacontane,	C_{30}
Tetracontane,	C_{40}
Pentacontane,	C_{50}

T = Trace

- **Basestock Type**

Basestock Type	wt%
Dibasic Acid Ester	
Polyol Ester	100
TMP Ester	
PE Ester	
DPE Ester	
Petroleum	
Synthetic Hydrocarbon	

Polyols	wt%
Trimethylolpropane, (TMP)	
Pentaerythritol, (PE)	100
Dipentaerythritol, (DPE)	

Mono Alcohols	
n-Heptanol	(C_7)
2-Ethylhexanol	(C_8)
Octanol	(C_8)
Nonanol	(C_9)
Decanol	(C_{10})
Undecanol	(C_{11})
Dodecanol	(C_{12})
Tridecanol	(C_{13})

TABLE F-12. BASESTOCK CHARACTERIZATION

SAMPLE DESIGNATION: K
AFLRL No.: AL-11266-L

Chemical Data

- Polyol Ester Components

Monocarboxylic Acids		wt%
Butanoic,	C_4	T
iso Petanoic,	C_5	
Pentanoic,	C_5	22
Hexanoic,	C_6	16
iso Heptanoic,	C_7	
Heptanoic,	C_7	24
iso Octanoic,	C_8	
Octanoic,	C_8	8
Nonanoic,	C_9	29
Decanoic,	C_{10}	1

- Dibasic Acid Ester Components

Dicarboxylic Acids	
Succinic,	C_4
Glutaric,	C_5
Adipic,	C_6
Pimelic,	C_7
Subaric,	C_8
Azelaic,	C_9
Sebacic,	C_{10}
--,	C_{11}
--,	C_{12}

- Synthetic Hydrocarbon Components

Hydrocarbon Type	
Triacontane,	C_{30}
Tetracontane,	C_{40}
Pentacontane,	C_{50}

T = Trace

- Basestock Type

Basestock Type	wt%
Dibasic Acid Ester	
Polyol Ester	100
TMP Ester	
PE Ester	99
DPE Ester	1
Petroleum	
Synthetic Hydrocarbon	

Polyols	wt%
Trimethylolpropane, (TMP)	
Pentaerythritol, (PE)	98
Dipentaerythritol, (DPE)	2

Mono Alcohols	
n-Heptanol	(C_7)
2-Ethylhexanol	(C_8)
Octanol	(C_8)
Nonanol	(C_9)
Decanol	(C_{10})
Undecanol	(C_{11})
Dodecanol	(C_{12})
Tridecanol	(C_{13})

APPENDIX G

INFRARED SPECTRA

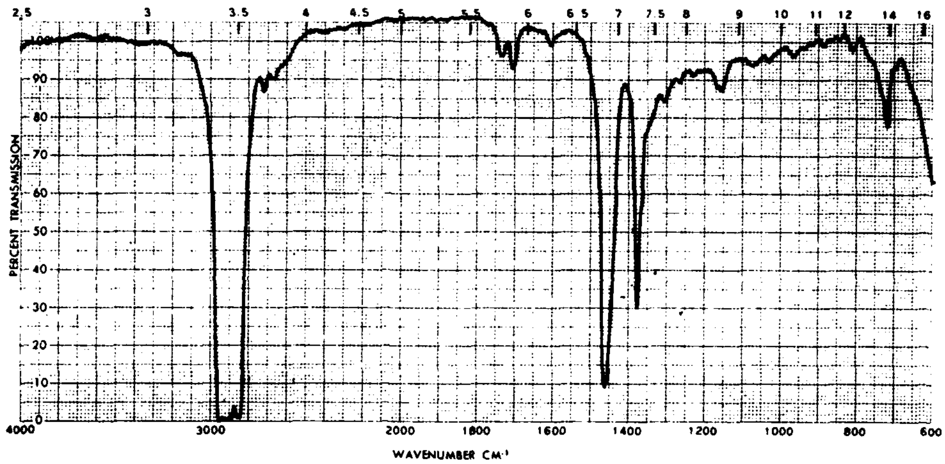


FIGURE G-1. NASA-A

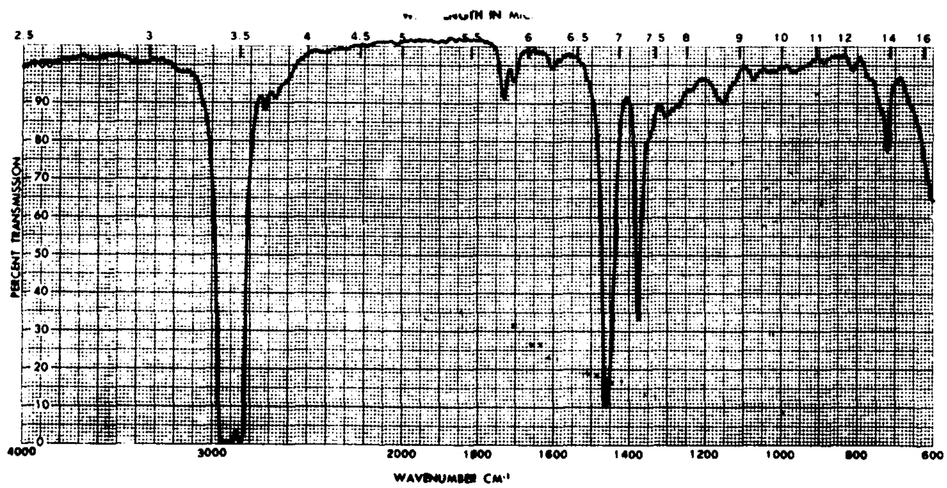


FIGURE G-2. NASA-B

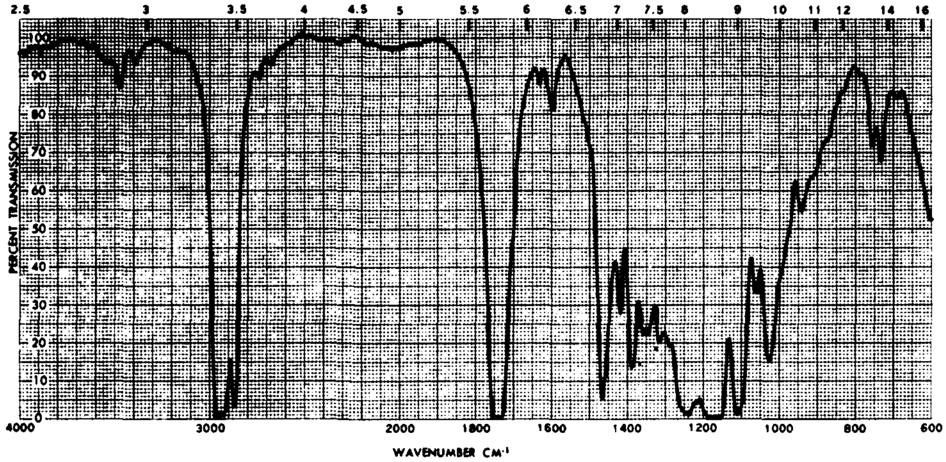


FIGURE G-3. NASA-C

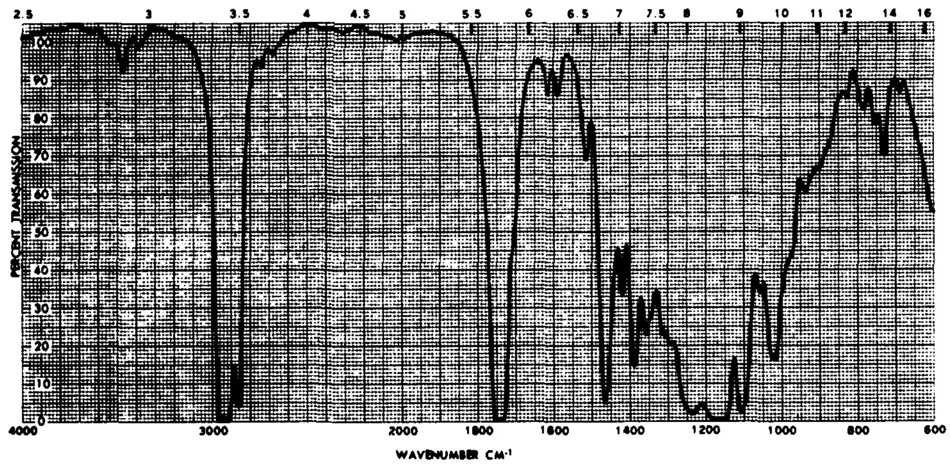


FIGURE G-4. NASA-D

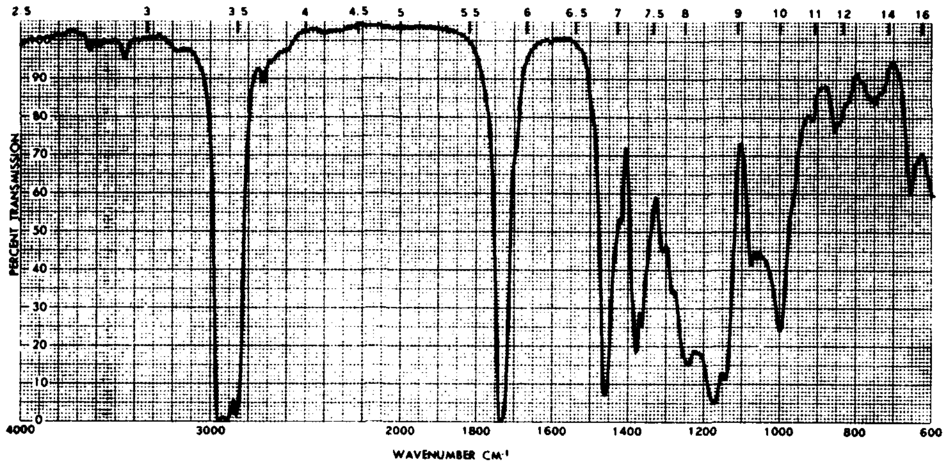


FIGURE G-5. NASA-E

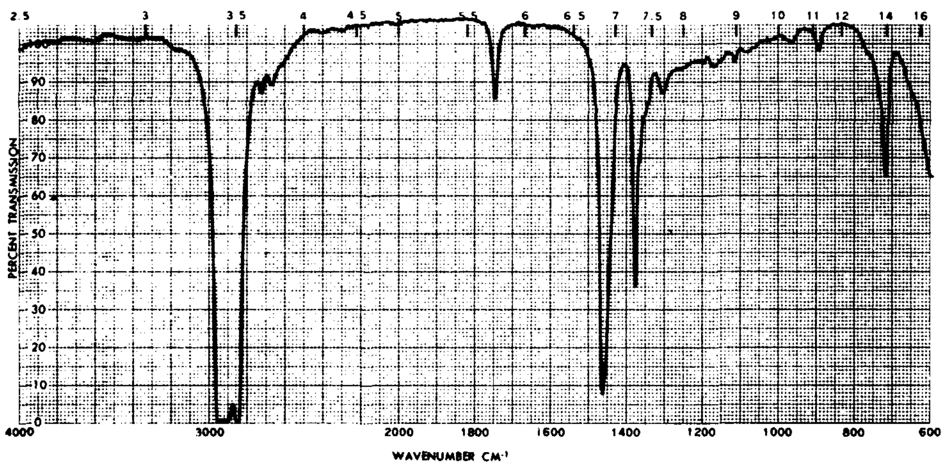


FIGURE G-6. NASA-F

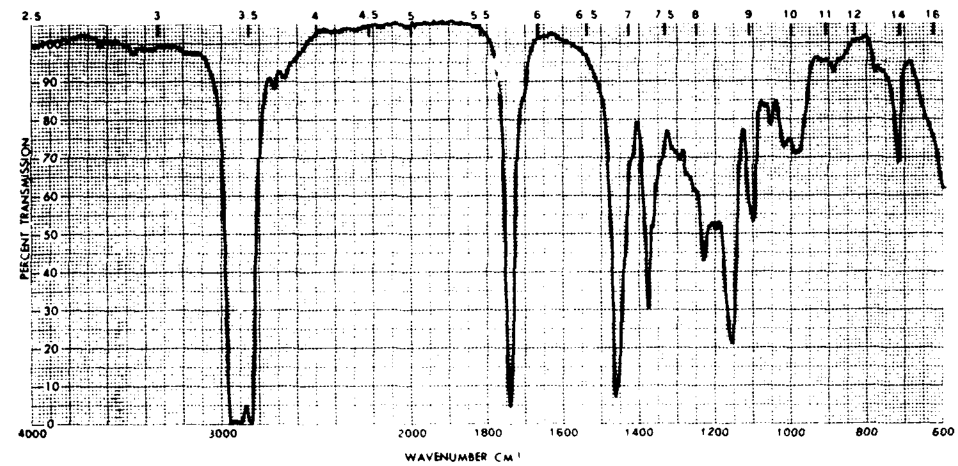


FIGURE G-7. NASA-G

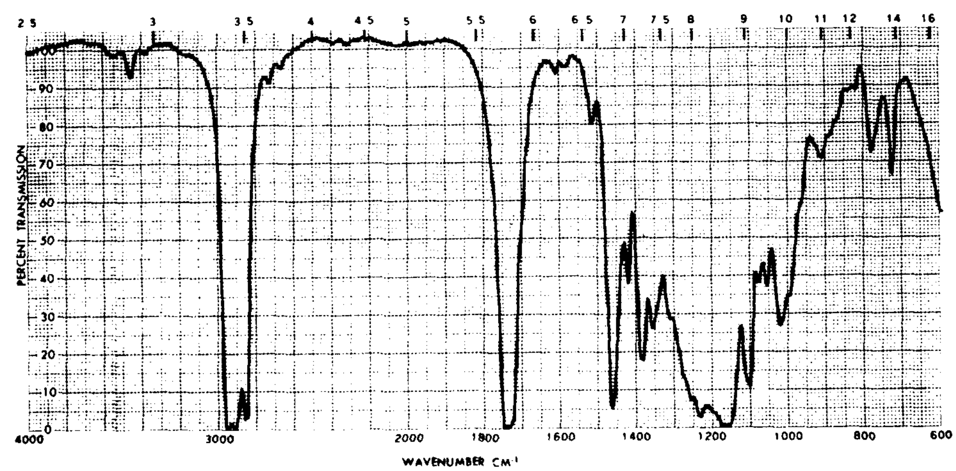


FIGURE G-8. NASA-H

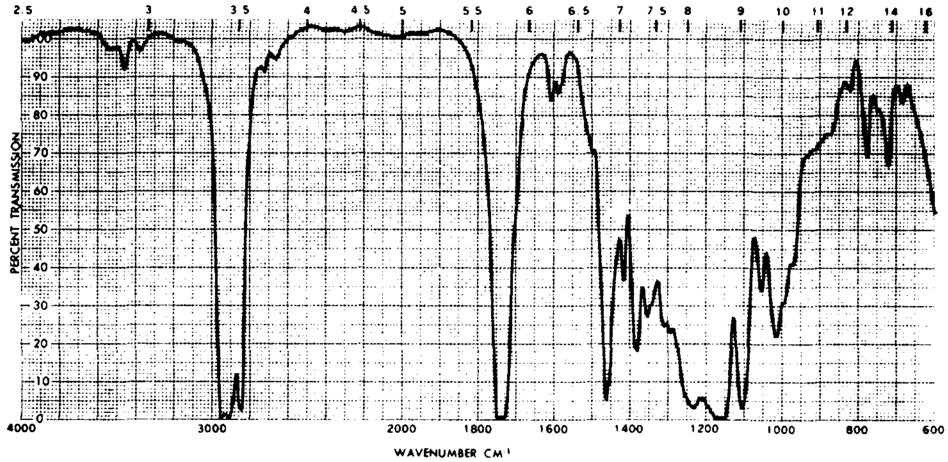


FIGURE G-9. NASA-I

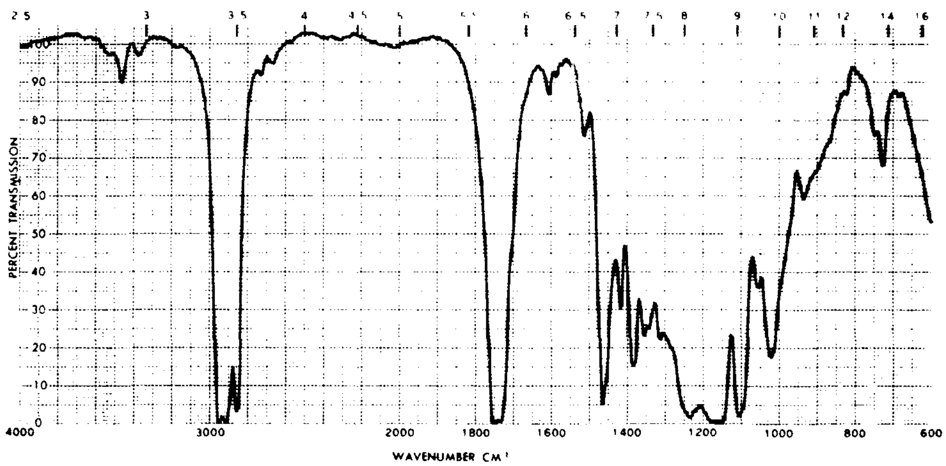


FIGURE G-10. NASA-J

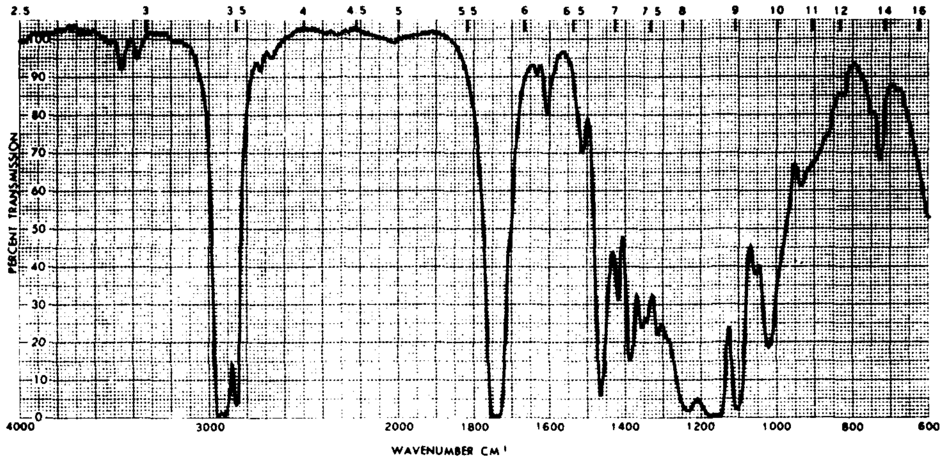


FIGURE G-11. NASA-K

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ATTN DRCPM-GCM-S 1
ATTN DRCPM-GCM-LF (MAJ SIKES) 1
WARREN MI 48090

PROJ MGR, FIGHTING VEHICLE SYS
ATTN DRCPM-FVS-SE 1
WARREN MI 48090

PROJ MGR, M60 TANK DEVELOPMENT
USMC-LNO, MAJ. VARELLA 1
US ARMY TANK-AUTOMOTIVE CMD (TACOM)
WARREN MI 48090

PROG MGR, M113/M113A1 FAMILY
VEHICLES
ATTN DRCPM-M113 1
WARREN MI 48090

PROJ MGR, MOBILE ELECTRIC POWER
ATTN DRCPM-MEP-TM 1
7500 BACKLICK ROAD
SPRINGFIELD VA 22150

PROJ MGR, IMPROVED TOW
VEHICLE
US ARMY TANK-AUTOMOTIVE CMD
ATTN DRCPM-ITV-T 1
WARREN MI 48090

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DRXMR-R 1
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DRDAR-SC 1
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BALLISTIC RESEARCH LAB
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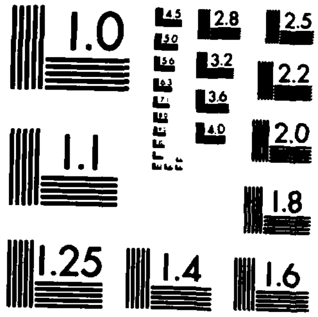

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ERRATA SHEET

Interim Report AFLRL No. 166
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"Advanced Chemical Characterization and Physical Properties of Eleven Lubricants" AD No. A131945 (CR-168187)

The NASA Code nomenclature for Figures A-1 through A-11 were designated incorrectly in the report. The correct codes are indicated in the right column opposite the erroneous designation.

APPENDIX A - PHYSICAL TEST DATA
(TO BE INSERTED BETWEEN PAGES 48 AND 49)

As Shown		Should be
FIGURE A-1.	NASA-A	Correct as is
FIGURE A-2.	NASA-B	NASA-F
FIGURE A-3.	NASA-C	NASA-B
FIGURE A-4.	NASA-D	NASA-C
FIGURE A-5.	NASA-E	NASA-D
FIGURE A-6.	NASA-F	NASA-H
FIGURE A-7.	NASA-G	NASA-E
FIGURE A-8.	NASA-H	NASA-G
FIGURE A-9.	NASA-I	Correct as is
FIGURE A-10.	NASA-J	NASA-K
FIGURE A-11.	NASA-K	NASA-J

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